Corrosion

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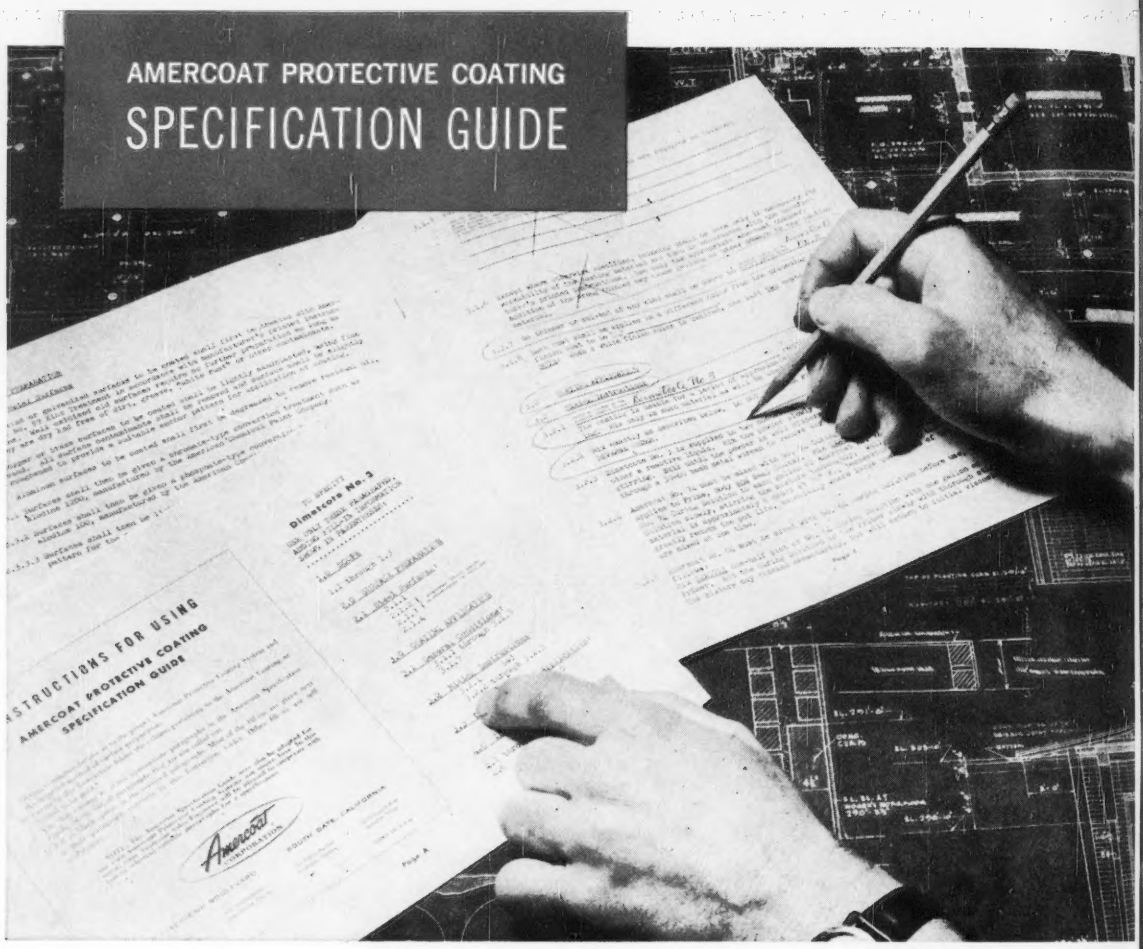
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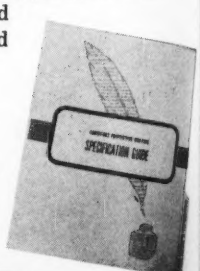
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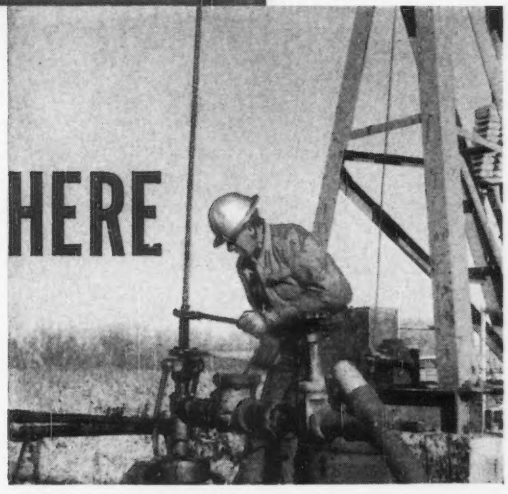
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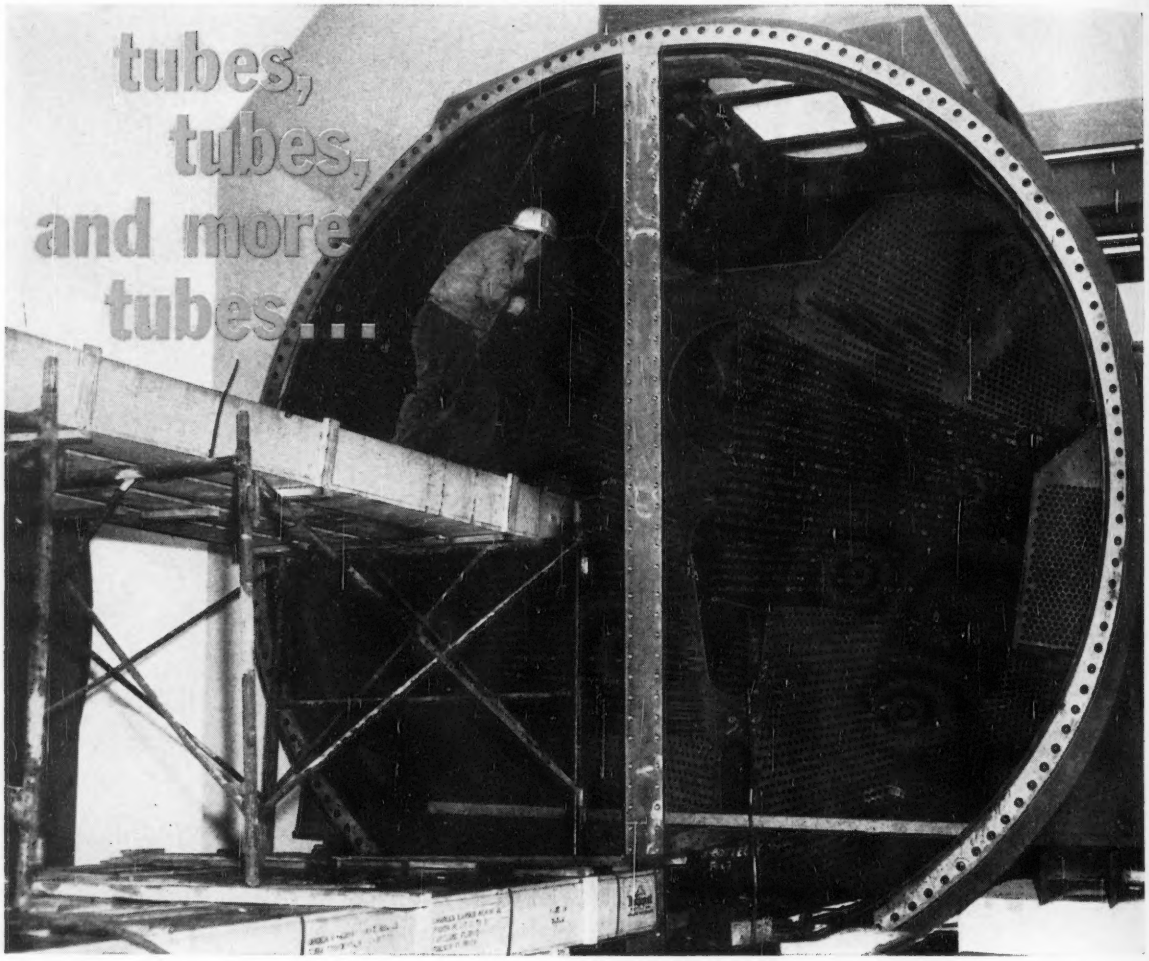
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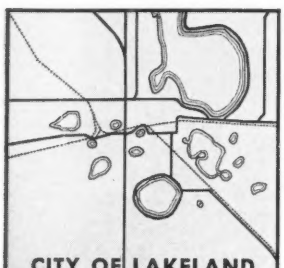
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This Month in Corrosion Control . . .

OIL SOLUBLE INHIBITORS can be used effectively to control corrosion in tankers and in pipelines. A thorough discussion of examples taken from actual service tests begins on Page 9. Interesting quantitative and economic data on this problem are mentioned.

PATCHING CORRODED EQUIPMENT in chemical plants with polyester and epoxy resins reinforced with glass has some advantages over other repair methods. Case histories of the method applied to steam chests, flaker hoods, pipe lines and other equipment are given in an article beginning on Page 22.

HIGH PURITY WATER environments found in atomic systems impose severe conditions on materials. Tests at the Bettis Plant and at Knolls Laboratory reported (Page 26) stress corrosion susceptibility of Type 410 stainless steel could be reduced by tempering at 1175 F.

PORTLAND NACE SECTION will hold the first short course on corrosion fundamentals ever held in Oregon April 27-29. See Page 34 for details. NACE has had some connection with more than 80 short courses in the last 10 years.

WHILE THEY NEVER BOTHER some people's stomachs, some foods are so severely corrosive that processing them is a strenuous test for materials. Recent experience shows that titanium clad vessels perform well in this service, as explained on Page 43.

NONUNIFORM RESISTIVITY conditions encountered in applying cathodic protection to underground structures complicated the task of protecting 21 sheath miles of lead sheathed telephone cable. How the problem was solved using an impressed current system is told in an article beginning on Page 83.

TROPICAL ENVIRONMENTS' influence on materials differ in several important respects from the effects of temperate zone conditions. These differences are considered in some detail, with data covering periods of exposure up to 8 years in the Panama Canal Zone, in Part 3 of a series starting on Page 87. Part 1 appeared in February and Part 2 in September, 1958. Copies are available.

MORE USEFUL DATA on the behavior of hydrogen absorbed in steel are given by three U.S. Steel Corp. workers in their article (Page 97) investigating the influence of cold work. As more and more information accumulates on this very important metallurgical phenomenon, science gets closer to some of the final answers.

IN A WIDE RANGE OF ACIDS found commonly in chemical plants, Type 202 stainless steel is comparable to Type 304 in all but the most strongly oxidizing environments. The article giving this information (Page 101) also shows that lean nickel grades' weldability is equivalent to that of Type 304. This 12-page article is filled with useful data and has 39 references.

CORROSION PRODUCTS literature is distributed in a large number of publications under a wide variety of subject headings. This makes the compilation of bibliographies on this subject by NACE T-3B particularly useful. Section Three of the series begins on Page 113. Other sections appeared in February and September, 1957.

ZIRCONIUM IN HYDROCHLORIC ACID under high pressures and at high temperatures corrodes in such a way that it is suspected that hydrogen has an important role. This reaction is studied in detail and with precision in an article starting on Page 118.

LIGHTNING PLAYS HAVOC with silicon rectifiers used to rectify for DC output. Data relating rectifier failure to a lightning incidence map, among other interesting facts about these devices, will be found in an article starting on Page 127.

SOIL BACTERIA find a better environment for activity at the bottom of a back-filled pipeline ditch than they find at equivalent depths in undisturbed soil, an extensive study beginning on Page 131 points out. This detailed investigation provides much useful information about bacteria that attack pipeline coating and wrapping materials.

PETROLEUM REFINERY CORROSION engineers attempting to determine the relative influence of variables in process streams on corrosion rates will find helpful practical information in an article on the use of statistical methods beginning on Page 137. Using an electrical resistance corrosion meter, plus statistical analysis, the writers develop a productive new method of corrosive evaluation.

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Cathodic Protection of Hulls and Related Parts, Technical Committee Report T-3G-1.

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AN EDITORIAL

Fallacy of Built-in Obsolescence

THOSE SEEKING weak links in the economy of the United States are prone to overlook what may be one of its most costly deficiencies. This deficiency, resulting from convictions based solely on sales goals, develops from the premise that products should be designed and fabricated to have built-in obsolescence.

Although this practice may be too strongly entrenched in the emotional aura surrounding the soft-goods industry to be attacked successfully, it is not necessarily immune to remedy when considered with respect to certain products which have, or closely approach, the status of capital investments. Some evidence strongly indicates that many intricate and often unreliable "sales point" differences built into such items as domestic applicances are put there solely to achieve distinction from other similar devices and are unnecessary, unneeded and result in a loss to the consumer. An equal amount of attention devoted to avoiding coupling of galvanically incompatible metals, to drilling drainage holes, to using durable coatings suited to the environments encountered, and to avoiding the use of flimsy, fragile components merely because they offer production economies would produce a far better product.

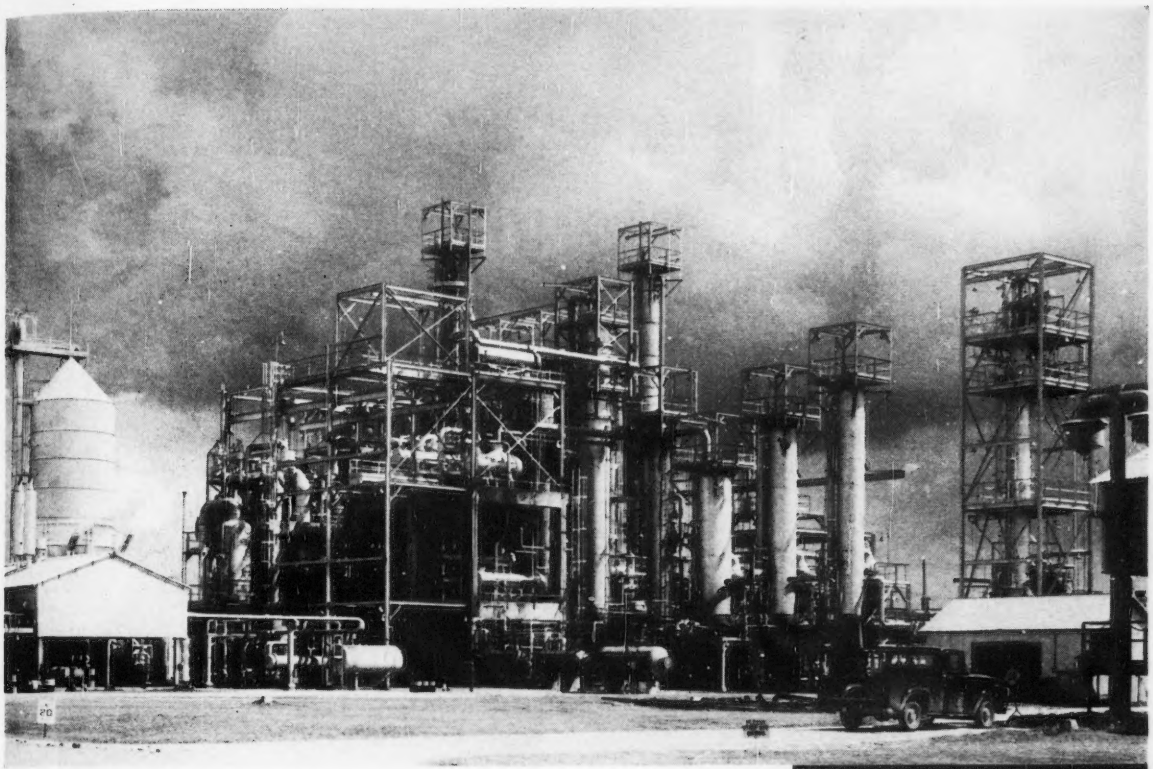
The most glaring example of obeisance to delusive consumer selling points is found in the automobile industry. Such elements as quadruple headlights, octuple tail lights, airplane-type fins; fantastically curved (and expensive) windshields; enormous, gas-gulping engines; yards of expensive chromium-plated bricabrac; excessively elaborate, expensive and frail radiator grills, among other features, serve to make the car more fragile and expensive while adding little, if any, to utility.

The automobile industry is geared to rapid unit turnover. It's sales goals contemplate a high percentage of trade-ins of cars still highly useful as transportation except for minor wear and such damage as they may have suffered from the elements and from collision. Millions wind up in dreary used parts yards in the environs of nearly every village and city. There they rust away year after year while their parts are cannibalized. Hundreds of millions of tons of steel, and considerable quantities of other metals, thus disintegrate into uselessly corroded metal which often cannot be reclaimed as junk.

This loss probably could be reduced materially if automobiles were designed and made to last indefinitely. Would it be better to sell fewer, more durable cars, and put the saved millions of tons of steel, glass and other metals they consume into other useful products? The automobile workers displaced could make other capital goods. The boom and bust character of the automotive market would be modified for the better.

The automobile industry has the experience, knowledge and ability now to make automobiles that will not rust unduly either inside or out. Some manufacturers already have taken the first steps to protect bodies. It can make engines of modest size, adequate efficiency, simple and with durable accessory components that will last far longer than today's engines. It can make tight, rattle-free, wind-proof, properly insulated passenger containers with comfortable seats. It can make strong, light, easily reached wheels, equipped with tires offering lengthy safe service life.

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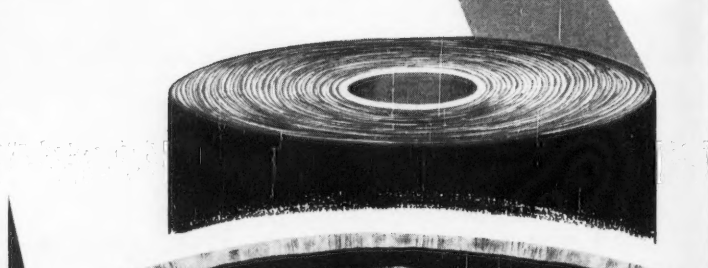
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Oil-Soluble Inhibitors for Controlling Corrosion in Tankers and Pipe Lines*

Abstract

Discusses causes of corrosion in the transportation and refining fields of petroleum industry where oil-soluble inhibitors can be used. Lists requirements of a good inhibitor. Gives a case history of use of inhibitor in one pipe line.

Refinery application of inhibitors is discussed briefly, also use of inhibitors in tanker ships carrying clean oil cargoes. Vapor space corrosion in tank ships is controlled by application of inhibitors by brush, flotation and fogging. Tests are discussed in which inhibitors were added to crude oil carried in tankers to control corrosion.

Corollary benefits of inhibitors added to petroleum products are given such as cleaner product, reduced wear in pumps, etc. Inhibitor's effect on product quality and inhibitor costs are briefly discussed.

Tabular data on tanker ship corrosion are given.

Introduction

APPLICATION OF oil-soluble inhibitors to prevent corrosion in the petroleum industry is discussed in this article. Comments are limited to major applications in the transportation and refining fields. Causes and background of corrosion in these fields are discussed and what some companies are doing to combat corrosion in a practical and economical manner is explained.

What are the results of corrosion in these fields? Rust clogs screens, plugs nozzles and jets in engines and oil burners and erodes meters, valves and pumps. It plugs filters in aircraft fuel handling equipment. Removal of rust from tank bottoms, ship tanks or products pipe lines is expensive. Loss of metal to rust often weakens tanks and lines, causing leaks, fires and other damage. It can cause serious product contamination aboard ship. It causes damage which must be repaired eventually if operations are to continue.

Corrosion losses are often over-looked because they may be listed as repairs or replacements. Cost of the direct loss of steel and its replacement due to corrosion is reported as six times the United States' national losses by fire. The loss to the tanker industry has been estimated at $150,000 per year per tanker.[1] A major United States pipe line reported several years ago that $800,000 per year were involved in reconditioning badly corroded lines. Another calculated a saving of almost $400,000 for a 250-mile products pipe line if corrosion could be controlled and a thinner wall pipe used with safety.

Before considering oil-soluble inhibitors, one should remember a few suggestions for means of reducing corrosion in these fields:

1. Replace steel with non-ferrous metal or plastic.
2. Keep oxygen away from steel by de-oxygenation, de-aeration or inert gas blanketing.
3. Keep water away from steel by barriers or coatings such as paints, inert oxides or iron complexes (as with chromate or nitrite water-soluble inhibitors) by dehydration of product or atmosphere or by invisible polar type monomolecular films of organic inhibitors. These all have been covered in the literature.[2-12] Only the use of oil-soluble organic inhibitors will be discussed here.

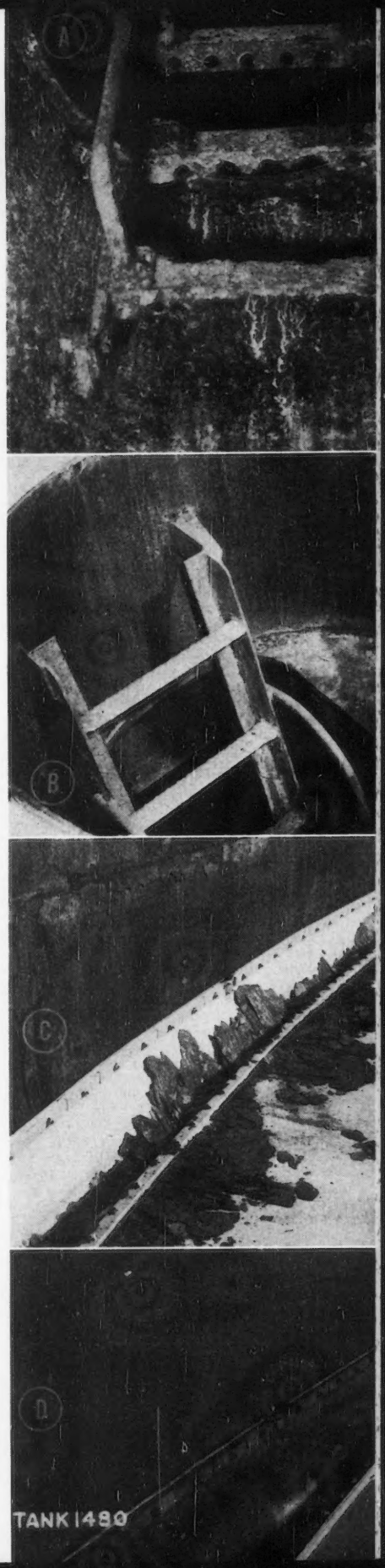
Requirements of a Good Inhibitor

A good inhibitor should have the following characteristics:

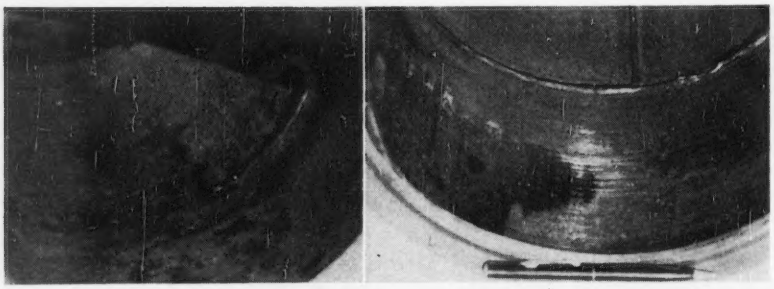
1. Be effective in preventing or reducing corrosion under various field conditions.
2. Be readily soluble in the product.
3. Have no adverse effect on other chemical or physical properties of the product.
4. Be free of deposit-forming ash.
5. Be permanent in its effectiveness and not be removed or rendered ineffective by handling or storage.
6. Be free of emulsification problems with water.
7. Be compatible with other inhibitors or additives (such as for corrosion, gum, dyes or tetraethylead).
8. Be non-toxic.
9. Be easily handled.
10. Be odorless.
11. Not seriously affected by traces of contamination resulting from normal refining or transportation handling.
12. Be insoluble in water.
13. Be low in cost.

(Continued on Page 10)

★ Revision of a paper titled "Oil-Soluble Corrosion Inhibitors" by W. S. Quimby, Texaco Inc., Beacon, N. Y., presented at the Canadian Region Conference, National Association of Corrosion Engineers, January 12-14, 1959, Montreal, Quebec.



CORROSIVE CONDITIONS and the successful corrosion control by use of oil-soluble inhibitors are shown. In Figure A, a typical rusty hatch and ladder are shown in an unprotected clean-oil tanker. Figure B shows a hatch area in a six-year old clean-oil tanker in which the area had been protected by brushing with oil-soluble inhibitor. Heavy scale flaking off the wall of a floating roof tank storing uninhibited gasoline is shown in Figure C. In Figure D, the wall of a floating roof tank storing rust inhibited gasoline has little or no scale.



FIVE-YEAR CONTRAST is shown between the corroded condition of the pipeline at left which had no internal protection and the relatively clean condition of the same pipeline at the right after five years of soluble inhibitor protection.

Oil-Soluble Inhibitors—

(Continued From Page 9)

Pipe Line Application

Water sometimes enters a pipe line suspended in the fuel because of improper settling in refinery tankage or due to improper handling of water bottoms in tankage. Water also enters dissolved in the fuel because the product generally is stored over water and is saturated to ambient temperatures. Upon cooling to ground temperatures, this water is precipitated and tends to wet the pipe wall. Turbulent flow in the line keeps free water from accumulating at the bottom of the line. The fuel is also saturated with air. Gasoline saturated with air at ambient temperature contains sufficient oxygen to produce 10 times the amount of rust removed by scrapers from an untreated pipe line.[13]

Oxygen and water in a pipe line cause rust. This rust is rough and increases the friction factor "C" and reduces pumping efficiency. This rust must be removed by a scraper, generally some form of a circular wire brush coupled to a pair of rubber cups which fit closely against the inside of the pipe line. Hydraulic pressure forces the scraper through the line. The rust is scraped free and pushed ahead of the scraper to the end of the line. Before the use of inhibitors became general, this scraping was required nearly every week. It was time consuming, costly and interfered with operations to a considerable extent.

On occasions, rust built up to the extent that the full 1000 pounds pressure would not remove the rust ahead of the scraper that became stuck and had to be located accurately, the line excavated and cut, the plugged scraper and rust accumulation removed and a new piece of pipe installed. This operation often took four days to complete during which the line was inoperative, sometimes several times during a year.

As an example of what inhibitors can accomplish, the following case history is typical. An old line was selected for a test with oil-soluble inhibitors. A commercial polar type of high molecular weight dibasic acid inhibitor was first used at 10 ppm. The "C" factor increased from 135 to 160-165 within a few months. Throughput and efficiency were restored to as-new conditions. Scrapers were run until most of the rust was swept from the line, after which the use of scrapers was practically discontinued.

When scrapers were run, they brought in no sediment. The stream was cloudy for a few minutes but settled clear in tankage in a few hours. Test sections of the pipe line removed for observation showed no trace of fresh rust after the first few months of inhibition. A single continuous injection of inhibitor provided essentially 100 percent rust protection in a pipe line for distances of at least 900 miles.

Inspection of product pipe line interiors using oil-soluble inhibitors showed that after the initial mill scale and rust formed during the laying of the line was removed by frequent scraping over the first few months, the lines operated at "C" factors of 155 to 160 and with only occasional token scraping. Test sections in these lines have shown complete protection over periods to six years. In one case, scratches to bright metal purposely made inside the pipe line have remained clean and bright for four years.

From a purchased inhibitor, Texaco switched several years ago to a corrosion inhibitor developed in its own laboratories. Present dosage in pipe lines is 6 lb per 1000 barrels. Results in all lines indicated complete freedom from rusting. Few, if any, scrapings were required, and the throughput remained at the initial design level. Corrosion prevention is reported to be much more simple and reliable than with other methods formerly used. A minimum of supervision is required.

Inhibition of aviation fuels has been a pipe line corrosion problem because U. S. military specification requirements originally did not allow such additives. Until performance characteristics of oil-soluble inhibited aircraft fuels were determined, commercial oil-soluble rust inhibitor was used by several pipe lines for several years under a temporary waiver of specification.

Most pipe line rust inhibitors now are added at the refinery where closer control of the addition can be maintained. In most cases, the inhibitor is added in the final fuel blending operation. This automatically provides the refinery with rust protection in its finished lines and tankage. This feature will be covered in more detail later.

The only problem encountered is when the blending stocks have not been completely freed of strong alkali or spent doctor. In these cases and when the tank bottom water is materially above a pH of 10, most inhibitors tend to form soaps which may be insoluble in the product and usually reduce inhibitor efficiency.

Laboratory and performance tests in pipe lines have demonstrated that inhibitors in use are compatible with each other in pipe lines. The author knows of no case where a product containing one oil-soluble inhibitor has had any effect on a product containing another rust inhibitor. In one system, five different inhibitors are being added, one by each company that uses the common line. This line reported that operations were satisfactory with no indications of internal rusting.

Similarly, tests and actual performance in pipe lines have demonstrated that inhibitors can be handled safely in water-soluble (sodium nitrite) inhibited pipe lines. There are several instances where inhibited products from one pipe line are routed through another sodium nitrite inhibited pipe line without any adverse effect upon the pipe line or upon product quality. Good rust protection, as judged by laboratory tests, is obtained at the end of the pipe line systems.

Although strong caustic will remove an oil-soluble inhibitor, a weak caustic such as that used in a sodium nitrite inhibited pipe line does not materially affect the inhibitor.

In lines without serious internal rust, a few parts per million of inhibitor in fuel is more than sufficient to create a monomolecular inhibitor film on hundreds of miles of pipe line and associated tankage. If heavy rust and deposits are present, inhibitor will be adsorbed on the surface of these materials. Spongy rust and scale presents a greater surface area than does clean steel, and the inhibitor is removed by adsorption as it tries to blanket the available surface. This situation continues for perhaps a few weeks until the rust becomes coated with inhibitor.

Use of scrapers is not necessary in pipe lines once initial rust and scale have been loosened and removed. There are numerous examples of old and new lines which were not scraped for at least four years, following the removal of loosened scale and heavy rust. When a scraper finally was run, no appreciable quantity of rust was removed, and only a slightly cloudy stream was obtained as the scraper was received at the end of the line.

The use of inhibitors in the protection of pipe lines is growing rapidly. Several pipe lines of major size have switched from dehydration[12] or water soluble inhibitor protection[2, 5] to oil-soluble inhibitors.

NACE Technical Unit Committee T-2E, Internal Corrosion of Products Pipe Lines and Tanks, is developing a proposed method for standardizing preparation and use of pipe line corrosion test coupons. This is aimed at a uniform method of simply and easily checking the effectiveness of pipe line inhibition. Table 1 shows typical monthly results on a pipe line using such a system for

(Continued on Page 12)

TABLE 1—Typical Monthly Report of Pipe Line Coupon Corrosion

	Rating*						
	Company					Main Line Pump Station	Terminal
Mo.	L	M	N	O	P		
Oct. 1957	A	B+	B+	B+	E	A	B++
May 1958	B++	A	B+	B+	B	B+	B++

* Explanation of Rating Scale:

A	Perfect. No rust.
B++	No more than 0.25 percent of surface rusted.
B+	0.25-5.0 percent of surface rusted.
B	5-25 percent of surface rusted.
C	25-50 percent of surface rusted.
D	50-70 percent of surface rusted.
E	75-100 percent of surface rusted.

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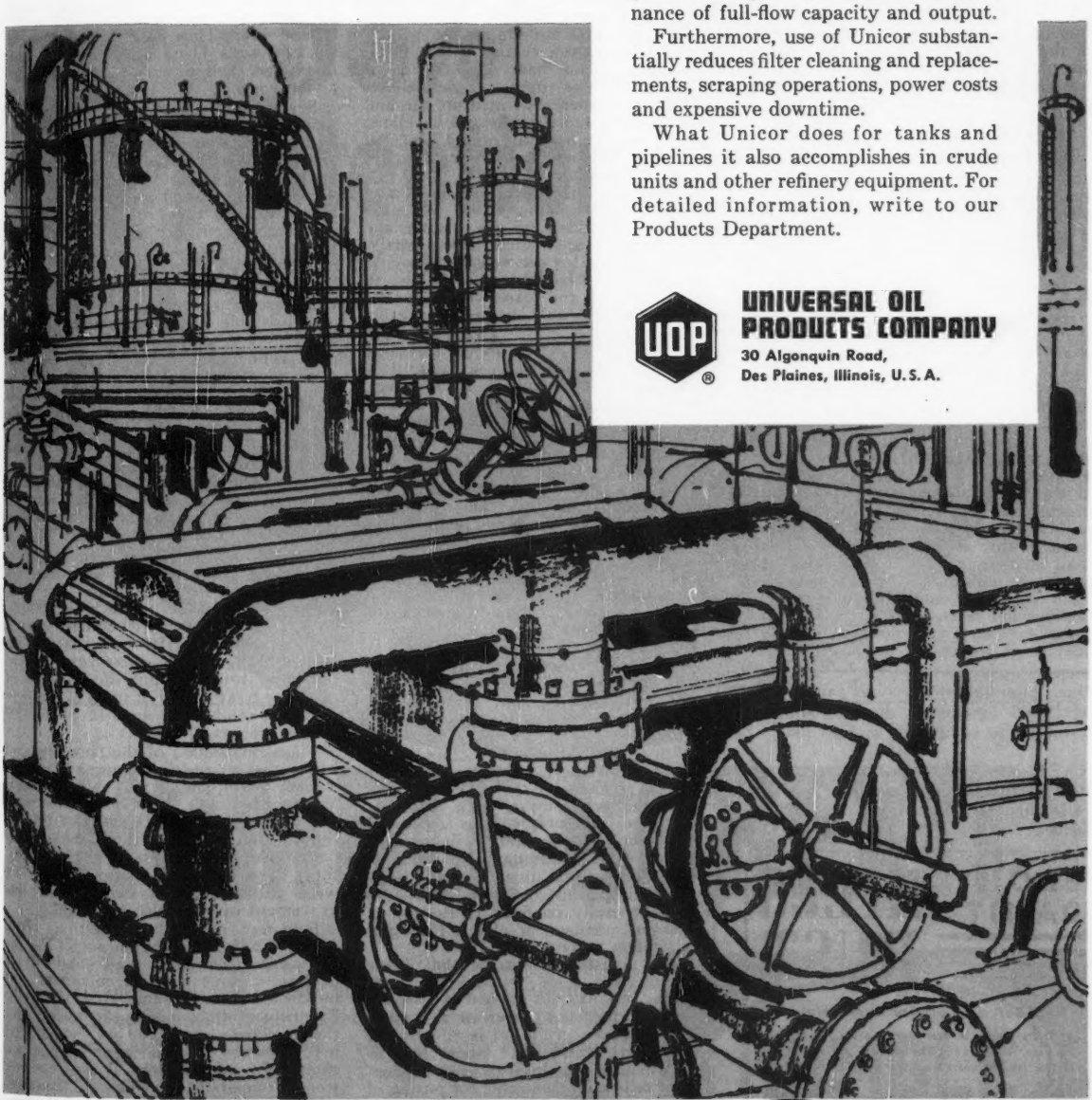
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Oil-Soluble Inhibitors—

(Continued From Page 10)

control. Test coupons of five companies supplying products to the line were installed in the pump station suction lines from these companies. The main line pump station coupon represented the pump discharge of the combined outgoing streams. The terminal coupon was used to monitor condition of the stream several hundred miles downstream on the incoming main line.

Ratings in Table 1 do not represent actual pipe line corrosion; that is, a "C" rating does not mean that 25 to 50 percent rusting is occurring in the pipe line. This is a sensitive test designed to show quickly if products having top quality rust protection are being handled. Each of these five companies uses a different oil-soluble inhibitor. Their acceptance was based on compatibility studies by the pipe line laboratories.

Refinery Application

Additional protection of tanks and lines can be obtained if the inhibitor specified for pipe line protection is applied in the refinery. This practice protects refinery equipment without additional cost. Tanks, particularly floating roof tanks, show less internal rusting when their contents contain inhibitors. For example, a survey of several refineries which use inhibitors has indicated that interiors of floating roof tank shells show less loose and falling scale, have a tighter adhering type of rust and generally have a darker, less active appearing rust than when inhibitors are not used.

An observation of a tank's interior shell will indicate whether or not inhibitors have been used in fuel stored in the tanks. Some inhibitors produce greater rust protection in tankage than others. This is probably to be expected because this exposure is more severe than that in ordinary pipe lines. Accordingly, differences in rust protection show up which might go unnoticed in the pipe line tests. Interior rusting is not completely arrested in floating roof tankage but is materially reduced. Benefits probably are of the fringe variety and by themselves might not justify the use of inhibitors. Addition of inhibitor during the final fuel specification blending, however, produces this degree of protection without noticeably affecting the inhibitive character of the fuels later.

The same is true of refinery finished product lines and manifolds. An inhibitor added to the fuel products refinery lines against serious rusting or replacement due to internal corrosion. The protective inhibitor film extends the life of these lines without extra cost.

Tanker Application

The average tanker of the T-2 size spends $150,000 per year to combat the direct and indirect effects of rusting.[3] More than 3000 tankers in the world[14] represent a sizeable rusting cost.

Tankers do not have double hulls or false bottoms as many people believe. The tanks themselves constitute the hull of the vessel, and the ribs and backbone of a tanker are her frames and keel. All structural members inside the tanks are exposed to the cargo.

Normally, ships' tanks are filled with cargo to the maximum permissible loading draft specified by law. After the cargo is discharged, some tanks, usually about one-third of the vessel's capacity, are loaded with sea water as ballast to insure stability and maneuverability. An empty tanker would be unmanageable in a sea way.

If the next cargo is to be the same that has been carried in a tank, it is pumped out and refilled. If, however, different cargo is to be carried, it usually is necessary to wash out the tank with sea water, usually at 150 pounds pressure, through a system of spray washing rotary nozzles. Often the water is heated to 180 F to speed removal of traces of cargo from tank walls. This combination of hot sea water at high pressures on steel in a marine atmosphere is very corrosive. Tankers operating in fresh water show negligible rusting. Tankers in fresh water service are still in good condition after more than 17 years without any corrosion protection.

In ocean service, however, empty tanks rust on the return voyage because of the salt atmosphere, water left in the tank after washing and condensation, particularly on the under sides of deck plating.

With clean cargoes (gasoline, kerosine, jet and diesel fuels and furnace oils), all portions of the tank rust with equal rapidity, resulting in a relatively uniform rusting surface. This is because the lighter products are easily washed off of the walls, leaving them prone to general rusting. With crude oil, however, rusting primarily consists of deep pitting generally confined to the top of horizontal members or plates. Pitting is in a random pattern.

Many investigators agree that crude oil itself is a good rust protective medium. In tanks not washed excessively with high pressure water or in empty but dirty tanks, crude oil is effective in preventing corrosion.[15] Some investigators believe that local oxygen concentration cells are responsible for pitting of washed tanks.

Unfortunately, some tanks must be washed with water to remove all traces of crude oil because ballast water carried must be discharged overboard in the open sea or in the loading port harbor. Unless the tanks are carefully washed, the discharged ballast would contain oil and create a serious pollution problem. There is strict governmental and international scrutiny for the pollution of coastal waters.

Clean Oil Tanker Application

Generalized rusting is severe when uninhibited clean oils are carried in tankers. Tanks and internal members last only 8 to 12 years before they must be replaced.[16] Heavy sheets of scale formed on all surfaces tend to trap cargo and cause contamination of subsequent cargoes. These scale sheets, sometimes 25 square feet, are a safety hazard to personnel. The scale must be knocked loose by high pressure water washing, scooped into buckets and hauled to the deck. As many as 300,000 pounds of rust have been removed by buckets from a tanker in one year. This washing and "mucking" requires considerable time and manpower and is a constant source of added costs.

Several methods to reduce this rusting have been proposed including coatings, de-humidification, de-oxygenation, inert gas blanketing, cathodic protection and water soluble inhibitors.[17–21] Each method has some merit and is being used commercially, but these methods generally are costly or require time and attention by the ship's crew in order to be satisfactory.

One method which is being used with considerable success is the application of oil-soluble inhibitors to tanker cargoes.[16, 22, 25] When the inhibitor is added at a refinery, a ship's crew need not conduct rustproofing operations, and no equipment or permanent investment is required aboard ship. The inhibitor's polar compounds form a dense invisible barrier against water as they do in pipe lines and refinery tankage. This film protects during the cargo voyage and also during ballast or empty return voyage. This phase of corrosion inhibition has been reported in the literature.[16, 23]

Several new ships engaged in coastwise clean products trade were used by one company in an inhibitor test program. These were modern 19,000-ton vessels with tanks of conventional design and construction. After five years of inhibited cargo service, these vessels were inspected to determine effectiveness of inhibitor treatment. For most of the five-year test inhibitor dosage of 24 lb/1000 barrels was used.

Visual inspection of the tank interiors below the cargo level showed that only a little hard, tight rust was present. Very little loose or falling scale was found. There were no blisters or pits on the internal structure. The tanks had a better than normal appearance. Actually they were rusty and rather rough but were judged to be a considerable improvement over uninhibited tanks.

However, physical observations often are misleading. Further proof of the inhibitors' effectiveness was obtained by drilling various internal members and bulkheads in these ships. Approximately 75 drillings were made each year, and the thickness measured by hook type micrometer to the nearest one-one thousandth of an inch. These readings were compared to original thickness measurements. Average corrosion rate was calculated as shown in Table 2. The indicated rate of 6.6 mils per year over the five year period is a considerable reduction over the original 14 to 16 mpy rate without any corrosion protection and to published industry figures of 25.6 mpy[23] and 23 mpy.[16, 24]

(Continued on Page 14)

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Oil-Soluble Inhibitors—

(Continued From Page 12)

Because shipboard measurements were considered variable and uncertain, specially prepared and weighed laboratory test coupons were installed on insulated tank racks on these inhibited, clean cargo vessels. After one and two year periods, the coupons were examined in the laboratory. The summarized data in Table 3 show that under inhibitor protection the corrosion rate in both ballasted and empty tanks is almost the same and averages 7.1 mpy for all coupons. This compares closely to the 6.6 mpy average for the test drilling method.

In addition to drilling and coupon measurements, a spot on the bottom of the access ladder in each tank was measured for thickness (after removal of rust and scale with a chipping hammer) each year. Typical results are shown in Table 4. The average rate of 4 mpy is a reasonable check on the previous values of 6.6 and 7.1 mpy.

Still another evaluation of rust protection was made by checking the amount of rust removed from tanks at cleaning periods. No previous history of rust removal was available on these ships, but other ships of smaller capacity operating in the same trade showed that from 150,000 to 200,000 pounds of rust could be reasonably expected annually between the fifth and tenth years of service. Table 5 shows the amount of rust removed each year for the cargo inhibited vessels. This amount of rust was much less than was experienced before cargo inhibitors were used.

Rust removed after inhibition was considerably finer than before inhibition. Some fine rust was carried out with the wash water during tank cleaning operations. Time required to clean up a normal vessel for shipyard (mainly in removing rust from tanks) was reduced. A cargo inhibited ship can be prepared for shipyard, including the mucking and gas freeing of all tanks, in 30 to 36 hours. Four or five days would have been required before cargo inhibitors were used.

Because of the reduced rust in inhibited tankers, the mucking procedure has been reduced to only once per year at the annual shipyard overhaul. Contamination of subsequent cargoes also has been reduced. The corrosion rate within a tank below the cargo level is approximately 4 to 7 mpy. Because the steel is 0.437 inches or thicker in these vessels and because steel is replaced generally when it has lost one-third its initial thickness, a useful life of over 20 years can be expected without major renewals. These data are in substantial agreement with those reported at 9.2 mpy for similar service.[23, 26]

With aircraft fuels, the use of oil-soluble inhibitors is limited to those specifically required by military specifications. Aircraft fuels, particularly aviation gasolines, are very corrosive to marine vessels, but for the present other means must be used to combat corrosion when carrying these products.

At least one major U. S. tanker operator tried oil-soluble inhibitors and found in laboratory and shipboard tests that corrosion was not appreciably reduced.[17] The large variation between these results and most others cannot be rationalized. The effects of tank washing and gas freeing are so great that care must be taken when comparing results reported by different tanker operators. Also, oil-soluble inhibitors vary in their effectiveness. Under certain conditions, the addition of any inhibitor to a cargo may be considered objectionable in the case of an independent operator hauling fuels for major companies.

Vapor Space Corrosion

Unfortunately, all corrosion on tankers cannot be controlled by addition of inhibitors to the cargoes. The vapor space above the cargo level is required by law for cargo expansion with rising temperatures. The tanks, therefore, cannot be filled with cargo. The vapor space, about 18 inches, is subject to condensation and is a great source of corrosion problems. Corrosion rates here have been as high as 40 mpy.

Application of a cargo soluble inhibitor has little effect on the under deck areas. Insufficient inhibitor is splashed onto the vapor space to provide protection. Laboratory and ship tests have shown that protection can be expected if sufficient inhibitor can be laid on the surface to be protected.

(Continued on Page 16)

TABLE 2—Five-Year Corrosion Rate on Ship Tanks With Inhibitors Used

Member	Average Rate, mpy
Shell Longitudinals	5.4
Transverse Wet Frames	4.5
Vertical Keels	1.8
Bulkhead Stiffeners	8.3
Stringers	7.6
Bulkheads	9.3
Shell Stringers	9.2
Average of members below cargo level	6.6

TABLE 3—Average of 1 and 2 Year Corrosion Rates on Internal Tanks With Inhibitors Used, Derived From Test Coupons*

	Average Rate (mpy)
Cargo + Ballast — center tank	9.0
Cargo + Ballast — wing tank	8.0
Cargo + Empty — center tank	5.5
Cargo + Empty — wing tank	6.0
Average, all tanks	7.1

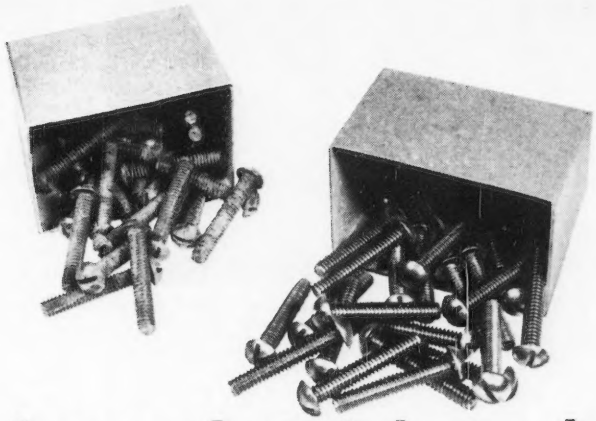
* Exposed at mid-tank depth.

TABLE 4—Corrosion Rates on Bottom of Ladder Exposed 5 Years to Inhibited Clean Cargo

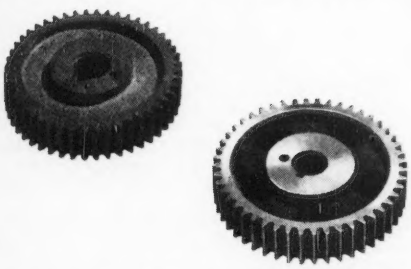
Tank No.	Rate (mpy)
1 Center	5
3 Center	3
4 Center	3
5 Center	2
6 Center	5
7 Center	4
8 Center	3
6 Port	5
7 Port	4
Average	4

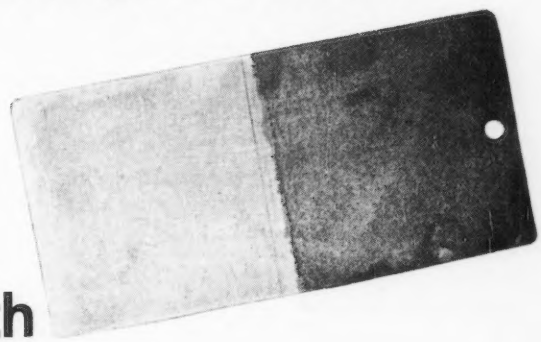
TABLE 5—Pounds of Rust Removed Annually From Tanks Carrying Inhibited Clean Cargoes

Year	Ship A	Ship B	Ship C	Ship D
1	40,500	48,700	54,800	40,400
2	44,100	64,800	56,400	58,300
3	43,800	59,300	36,500	42,600
4	56,600	79,600	55,700	50,700
5	56,100	53,000	57,200	.....
Annual Average	48,200	61,100	52,100	48,000



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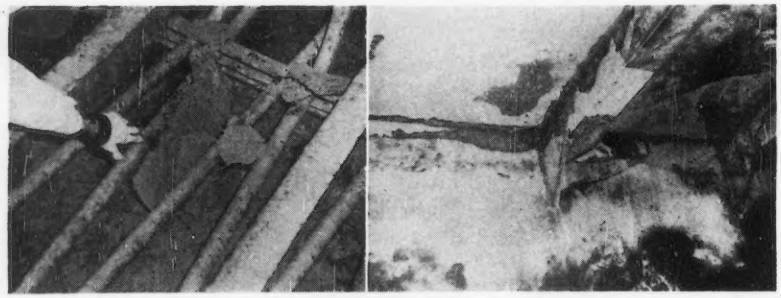
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OIL TANKER CORROSION is illustrated at left by the corroded areas and large chunks of scale which fell in an unprotected tanker. At the right is a typical rusty under-deck space of a clean-oil tanker protected by oil-soluble inhibitors alone, which did not reach the upper areas inside the tanker. These areas must be protected by fogging the inhibitor onto the surfaces not in contact with the inhibited product being transported.

Oil-Soluble Inhibitors—

(Continued From Page 14)

Inhibitor Application by Brush

The first place to be protected was the hatch coamings and covers. Here the rust and scale flake off rapidly; replacement of the top ladder rung often is necessary in a few years. The first inhibitor application in these areas was by brushing, each time the vessel was in ballast. Trials were made on rusty hatches and on hatches which had been chipped to clean metal.

After one year, rusted areas were scale-free with only a hard, tight and inactive rust film. The de-scaled hatches were covered with a similar light rust film. These results were so satisfactory that this scheme was adopted on all company vessels. On some vessels, the inhibitor was applied by paint spray gun rather than by brush with the same protective results. Over a five-year period, rusting has been practically arrested. The chipping hammer marks were still faintly visible after three years with this form of protection. No measurements of rusting rates were obtained on the hatches or coamings.

Flotation Application

From laboratory trials and small scale tank tests, the most practical way to protect the under deck areas of ballast tanks was by flotation.[2] The inhibitor was dumped on the rising ballast water and the thin, oily layer floated upward into contact with the under deck members by slowly filling the tank to capacity. This is known as "pressing up" the tanks. The amount of inhibitor used was determined to be approximately two gallons per 1000 barrels capacity. The amount of inhibitor remaining after the ballast was discharged was considered negligible and could not be detected in subsequent cargoes containing no inhibitor such as military specification aviation or jet fuels.

Visual observation after one and two years of flotation tests showed the under deck area was almost free of all scale with the same hard adherent rust film noted on the hatches. The vapor space areas appeared to be in as good condition as the tanker members below the cargo level.

To supplement visual evidence, test coupons also were exposed in these areas. Ladder rails in the hatch coamings (and above the cargo level) and deck supports were measured for actual wastage. In a two-year test, the upper ladder corrosion rate in unprotected tanks averaged 33 mpy, which is comparable to similar measurements on other vessels. In flotation treated tanks, the rate was only 5 mpy or an 85 percent reduction in corrosion. Table 6 shows corrosion rates on laboratory coupons exposed under deck. These observations confirmed that under deck corrosion of ballast tanks can be reduced by an oil-soluble inhibitor applied by the flotation method. This method is now being used on all clean oil as well as crude oil tankers.

Application by Fogging

The under deck corrosion of non-ballast (empty tanks) was still a problem. Based on laboratory and small scale tank tests, a trial aboard ship was made of inhibitor application in empty tanks by the fogging method. A finely atomized spray or fog of the inhibitor concentrate in air or steam covered all points of the under deck area. Surprisingly, even areas at a considerable distance from the fog nozzle and not facing it were protected.

A simple fog system was installed experimentally in several tanks of a clean cargo ship that was three years old and rusty under deck in the normally non-ballast tanks. Rust inhibitor was introduced at 2 gallons per 1000 barrels of tank capacity directly into a small steam line. The mixture of partially vaporized inhibitor was piped directly into the upper part of the tank. The injection was about two feet below deck at four equally spaced locations. No special nozzles were found necessary. A regular ¼-inch by ½-inch reducing bushing facing upward toward the under deck surfaces was a satisfactory nozzle. The fog treatment was applied for 10 minutes as soon as practical after the cargo had been discharged after each voyage.

After one and two-year trials in empty tanks, inspections indicated the under deck areas above the normal cargo level to be free of loose and falling scale with only a thin adherent coating of rust. The appearance was similar to the ballast tanks protected by the flotation method.

The same type of deck support and ladder measurements and test coupon exposure tests were conducted as described for the flotation method. In the fogged tanks, the ladder measurement showed an average corrosion rate of only 4.3 mpy compared to 21 in comparable nonfogged tanks. Table 7 shows typical data for laboratory coupons exposed during these fogging tests.

These observations with fogging confirmed that serious corrosion of under deck areas can be prevented by fogging with an oil-soluble inhibitor. The laboratory coupon data for the wing tank under-deck corrosion are low in Tables 6 and 7. This cannot be explained except by postulating that the insulated coupons did not receive the same degree of condensation as members directly in contact with the deck plating. This would indicate that the coupons received sufficient protection from splashing of the inhibited cargo. The corrosion rate is in line with values obtained below the cargo level. Because of the excellent results obtained in the two-year trials, complete fogging systems for all tanks on additional vessels are under construction.

Crude Oil Tanker Application

Deep pitting corrosion on crude oil tankers has been so severe that some ship bottom plates required replacement in four to six years and the heating coils (of thinner material) in two to four years. Most commercial methods for preventing corrosion in these vessels were quite expensive to install and maintain.[15]

Tests were conducted to see if inhibitors could be used in crude oil vessels. Laboratory tests were favorable and tests were begun on several 28,000-ton tankers carrying Arabian crude, a portion of which was carried subsequently via pipe line.

A polar type oil-soluble inhibitor was added directly to the crude as it came aboard ship. A simple pressurized inhibitor reservoir was installed with the injection rate controlled by a valve in accordance with the crude loading rate. Inhibitor concentration during most of this testing was 24 lbs per 1000 barrels of crude oil. The ships were three years old and had experienced considerable pitting on the bottom plates, cargo piping and heating coils. Annual inspections were made for six years following the inhibitor application.

Bulkheads and internal members showed only negligible uniform corrosion. In many cases, small patches of mill scale were detected on vertical surfaces. Horizontal surfaces, particularly the heating coils, showed characteristic random pitting. Compared to the initial rate of pitting over the first three years and with competitive ships operated under the same conditions, the vessels showed considerable reduction in pitting corrosion. In many cases, pits appeared to be inactive; others continued to corrode.

A few pits were 0.700 inches deep.

(Continued on Page 18)

TABLE 6—Corrosion Rates on Laboratory Coupons Exposed 2 Years in Flotation of Ballast Tanks

Tank Condition	Corrosion Rate (mpy)	
	Center Tanks	Wing Tanks
Ballast—Average	20	8.5
Ballast+Flotation—Average	4	2

TABLE 7—Corrosion Rates on Test Coupons Exposed 2 Years to Fogging of Inhibitors in Empty Tanks

Tank Condition	Corrosion Rates (mpy)	
	Center Tanks	Wing Tanks
Empty—Average	23	6
Empty+Fogging—Average	4.2	6

FACT: In salt water test 4-D Wrought Iron proves 39% more corrosion-resistant than A-53 Steel

The fact stated above is the result of a recently completed corrosion test conducted at A. M. Byers Company Laboratories. The test lasted a full year.

These results showed A-53 Steel weight loss to be 39% greater than 4-D Wrought Iron over the 12-month period. In addition to this substantial difference in weight loss, 4-D Wrought Iron did not exhibit pitting. After only three months, pitting appeared on the steel. In the final months of the test, pits on steel had expanded and consolidated until they appeared as drastically thinned areas.

In this test, exposed coupon ends and edges comprised as much as 20% of the total coupon area. Under actual service conditions, however, ends and edges are not exposed or comprise but a minute fraction of the total exposed surface.

Since wrought iron displays directional properties with regard to corrosion-resistance, a corollary 9-month test was conducted with a coating on the edges and ends of all test coupons, steel and wrought iron. *This* test showed 44% greater corrosion-resistance for 4-D Wrought Iron over A-53 Steel. Like the 12-month test, results substantiate 4-D Wrought Iron's corrosion-resistance superiority over steel.

TEST CONDITIONS

Test medium:	3% sodium chloride solution
Test temperature:	68° to 75°F. (average: 70°F)
pH:	Average 7.8
Aeration rate:	3400 cc/min±10%
Solution volume:	32 liters changed three times each week
Coupon size:	1″ x 2″ x ⅛″ or ¼″
Coupon preparation:	Solvent degreasing and polishing on belt grinder using 100-grit carborundum belts
Test period:	1. One year 2. Nine months with 4-D and A-53 Steel coupon edges coated

SUMMARY

The results of both salt water tests establish 4-D Wrought Iron's superiority over A-53 Steel in corrosion-resistance. These results are verified by hundreds of installations where corrosion is a constant problem; and, where wrought iron has performed satisfactorily for years at low cost per year of service. Write for complete test report. A. M. Byers Company, Engineering Service Department, Clark Building, Pittsburgh 22, Pennsylvania.



BYERS 4-D WROUGHT IRON

TUBULAR AND FLAT ROLLED PRODUCTS

Oil-Soluble Inhibitors—

(Continued From Page 16)

After nine years of crude service (six years being in inhibited crude) most pits ranged from 0.300 to 0.600 inches deep and were on the upward facing horizontal surfaces. Bulkhead pitting or blistering was practically nonexistent. The average corrosion rate was small. Deep pitting was repaired by welding or by plate removal to avoid leakage and creation of an unsafe condition.

Relatively little rust was removed when the tanks were mucked. Most material removed was a waxy residue which tended to settle from the crude oil. The corrosion rate was quite low, based on visual observations and a thickness measurements of critical members.

Use of an oil-soluble inhibitor reduced the pitting corrosion but not to an acceptable value. These vessels may require considerable welding or plate replacement before the end of 12 years of service.

Pits which were present initially or which formed during the test tended to hold water upon the discharge of the cargo and after washing, thus preventing the inhibitor in the crude from reaching the underlying steel. If inhibitors could be applied to dry tanks before loading, results might be better. The inhibitor treatment method is being continued in conjunction with tests on coatings and galvanic anodes in hopes that an improved system can be established to control pitting corrosion.

Corollary Benefits

Corollary benefits of oil-soluble inhibitors in clean product lines and tankage are obtained by protection of the internal walls of terminal and bulk plant tanks, tank cars, trucks and underground storage tanks. Inhibitors in fuels have reduced wear on service station pumps due to the absence of excessive fine rust.[3] Fuel soluble inhibitors are reported to reduce clogging of screens in equipment by preventing formation of rust. At least one claim has been made that corrosion of automotive equipment can be reduced by oil-soluble inhibitors.[4]

Effect on Product Quality

In the low concentration used, oil-soluble inhibitors do not materially affect the quality or the usual physical tests of products to which they are added.[16] Many companies have been using inhibitors for over 10 years in refined products without incident. Laboratory and engine tests have shown that automotive and aviation fuels are not degraded in any manner. The same is true of kerosine, diesel fuel and furnace oil.

These corrosion inhibitors have been demonstrated to be compatible with all other fuel additives that may normally be present to control gum, color, oxidation, combustion or icing. Many pipe line specifications list acceptable oil-soluble corrosion inhibitors. U. S. military specifications for aviation fuels require corrosion inhibitors.

Costs

The cost of inhibitor application for corrosion protection is difficult to determine. Inhibitors vary in purchase price, in freight charges to destination, in dosage required for protection and in application costs. Generally, the high direct and indirect replacement costs caused by corrosion more than justify the use of inhibitors. In isolated cases such as short movements by pipe line or tanker, the unit cost per barrel mile can become excessive, and other means such as water soluble inhibitors or coatings can be substituted. Most applications of oil-soluble inhibitors are reported to be the most economical method found for the degree of protection obtained.

Conclusions

The applications of oil-soluble inhibitors discussed suggest the following conclusions: They are effective in reducing maintenance costs due to corrosion in the storage and movement of refined petroleum products and are partially effective in reducing the specialized pitting corrosion in crude oil tankers. Effective in a single application and easily applied, inhibitors are permanent in their protective quality because inhibited products retain their rust protection in subsequent handling until consumed. They are effective in very low concentrations and have no appreciable effect on product quality. They are effective for direct application to steel surfaces not in contact with liquid petroleum products.

Acknowledgment

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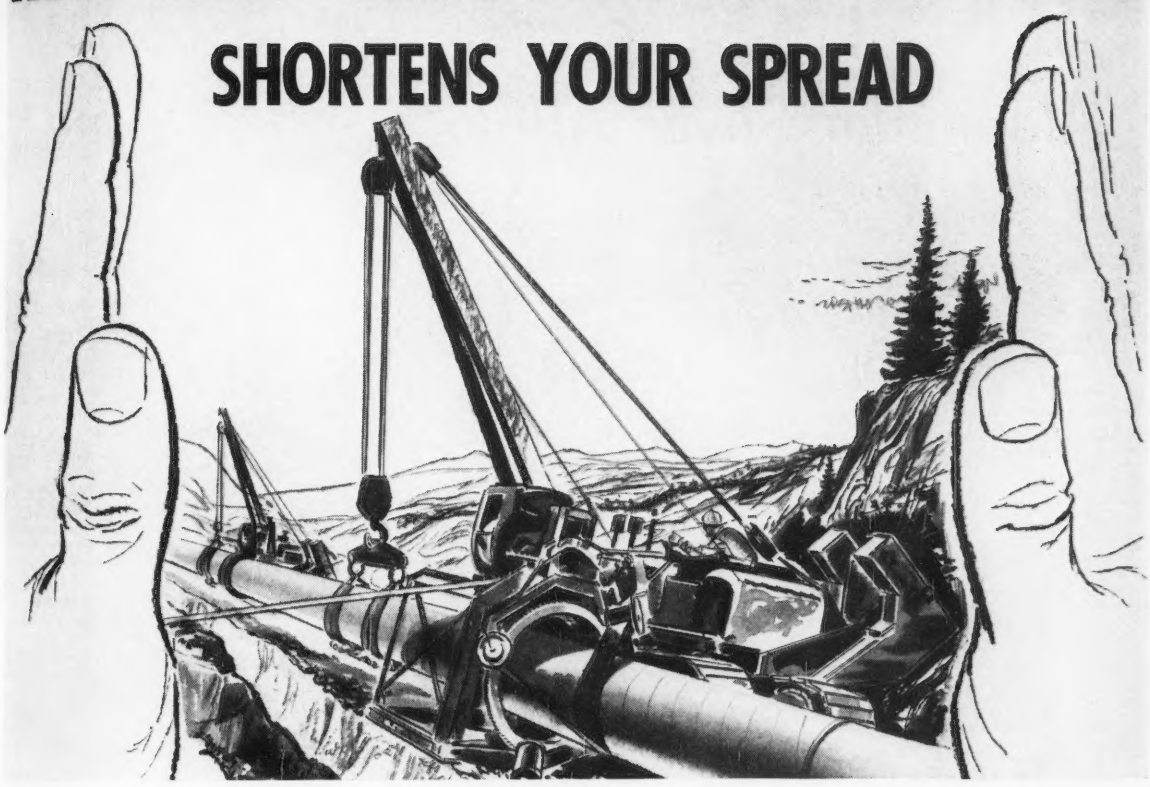
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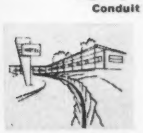


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Accelerated Corrosion in Aluminum-Silver Alloys*

Abstract

Atmospheric corrosion of aluminum-rich aluminum-silver alloys from which Ag_2Al was precipitated led to complete pulverization of samples within a few weeks. In this process the intergranular intermetallic compound corroded out completely. Corrosion proceeded at a mild rate in an ordinary atmosphere but became catastrophic with high humidity.

6.4.2

Introduction

ALUMINUM - SILVER ALLOYS which disintegrated completely in atmospheric exposure are discussed in this article. The samples were stored in a refrigerator at 6 C. After four weeks, they had disintegrated into a brownish powder. Samples with lower silver contents had a more granular appearance. No sample of less than 25 weight percentage silver was pulverized completely.

Because these alloys were of the precipitation hardening type (maximum solubility 48 weight percentage silver at the eutectic temperature), this catastrophic corrosion attack was attributed to the precipitated phase. The milder attack on alloys with lower silver contents was related to smaller amount of precipitate or to the lower silver content of the solid solution or to both. This corrosion was considered unusual; other researchers working with these alloys had not observed such corrosion before.

★Revision of a paper by H. H. Stadelmaier, North Carolina State College, Raleigh, N. C., and E. M. Whitener, Idaho Chemical Processing Plant, Phillips Petroleum Co., Atomic Energy Division, Idaho Falls, Idaho.

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Environment

Samples containing 40 weight percentage silver were tested to determine the nature of the environment conducive to pulverization. Exposure to the laboratory high humidity atmosphere during the summer produced noticeable surface attack. Storage in dry argon for four weeks produced no trace of attack. Corrosion is indicated by the complete absence of a reaction in the dry atmosphere. Exposure to an atmosphere of 100 percent relative humidity accelerated the reaction to the extent of beginning pulverization within 24 hours.

To determine whether the reaction was sensitive to impurities, the original alloys consisting of low grade aluminum (97.2 percent pure) and fine silver (99.96 percent) were replaced by alloys of high purity aluminum (99.996 percent) and silver (99.997 percent) which were melted in a crucible of fused alumina. The high purity alloys corroded essentially as rapidly as the original ones.

Two factors related to the storage in the refrigerator appear significant:

1. The refrigerator was opened frequently, allowing entry of air of temperatures around 30 C and relative humidities around 90 percent during the season in question. This caused frequent condensation of water on the cold surfaces inside the refrigerator and also on the specimens.

2. Some concentrate of photographic developer (Metol-hydroquionone type) was stored in the refrigerator and was in current use. Exposure of a new sample to an atmosphere saturated with the volatile developer components showed that the corrosion attack was accelerated in the presence of the developer. In view of the experience with humid air, it must be assumed that the role of the developer is secondary.

Corrosion Process

X-ray diffraction analysis of the corrosion products representing three 40 percent silver specimens solution treated at 550 C and left to precipitate at 450 C (2 hours), 380 C (3 hours) and 300 C (12 hours) respectively showed aluminum oxide hydrate, aluminum solid solution and silver. Surprisingly, diffractions of Ag_2Al were absent from the corrosion products whereas those of the aluminum-rich solid solution were still present. The phase diagram indicated that at 40 weight percentage silver the ratio of Ag_2Al to Al is nearly one to one. Therefore it must be concluded that the intermetallic compound was preferentially attacked. This is unusual because Ag_2Al has a cathodic corrosion potential with respect to aluminum at pH values ranging from acid to alkaline. In metallographic etching with sodium hydroxide, it is the unattacked phase. Also, the corrosion potential of Ag_2Al against the (homogeneous) solid solution at 40 weight percentage silver was cathodic in the entire range of pH values. The compound was anodic only with respect to silver.

Microscopic study of the early corrosion stages showed specks of a black corrosion product appearing after one day in ordinary room atmosphere. This corrosion was adjacent to the larger particles of Ag_2Al in the grain boundary region, suggesting that the process begins in the familiar manner by galvanic corrosion of solid solution that is depleted in silver and therefore most anodic. Next the attack spread to the large particles of Ag_2Al, which were covered with the dark corrosion product after five days in the room atmosphere. This confirmed the diffraction data indicating that the compound Ag_2Al was preferentially attacked and gave the corrosion an intergranular appearance. Finally, the whole grain interior was covered only after a period of weeks. Because of this destruction of the grain boundary material, pulverization of the alloys was inevitable.

Unlike the behavior in Al-Cu alloys,[1] the corrosion did not stop in the depleted region but also involved the compound. This can be seen best by studying the behavior of a homogenous alloy of the composition near Ag_2Al. It remains practically uncorroded after months in air of moderate to high humidity but shows extremely severe corrosion after 12 hours in an atmosphere of 100 percent relative humidity. This reaction is accompanied by the eruption through the surface of corrosion products giving the whole surface a blistered appearance.

Galvanic corrosion of Ag_2Al has to involve silver as cathode. It is not difficult to show that elemental silver is one of the corrosion products in both Ag_2Al and the original alloys of 40 weight percentage silver. Thus the corrosion process shifts from the depleted solid solution to the intermetallic compound instead of the more anodic grain interior because the compound has a demonstrated tendency to corrode, which is much greater than that of the grain interior. (The grain interior corresponds to the low-silver alloys that did not corrode severely.) This preferred attack of the compound is probably connected also with the absence or continued disruption of any protective layer of corrosion products on the compound.

Acknowledgments

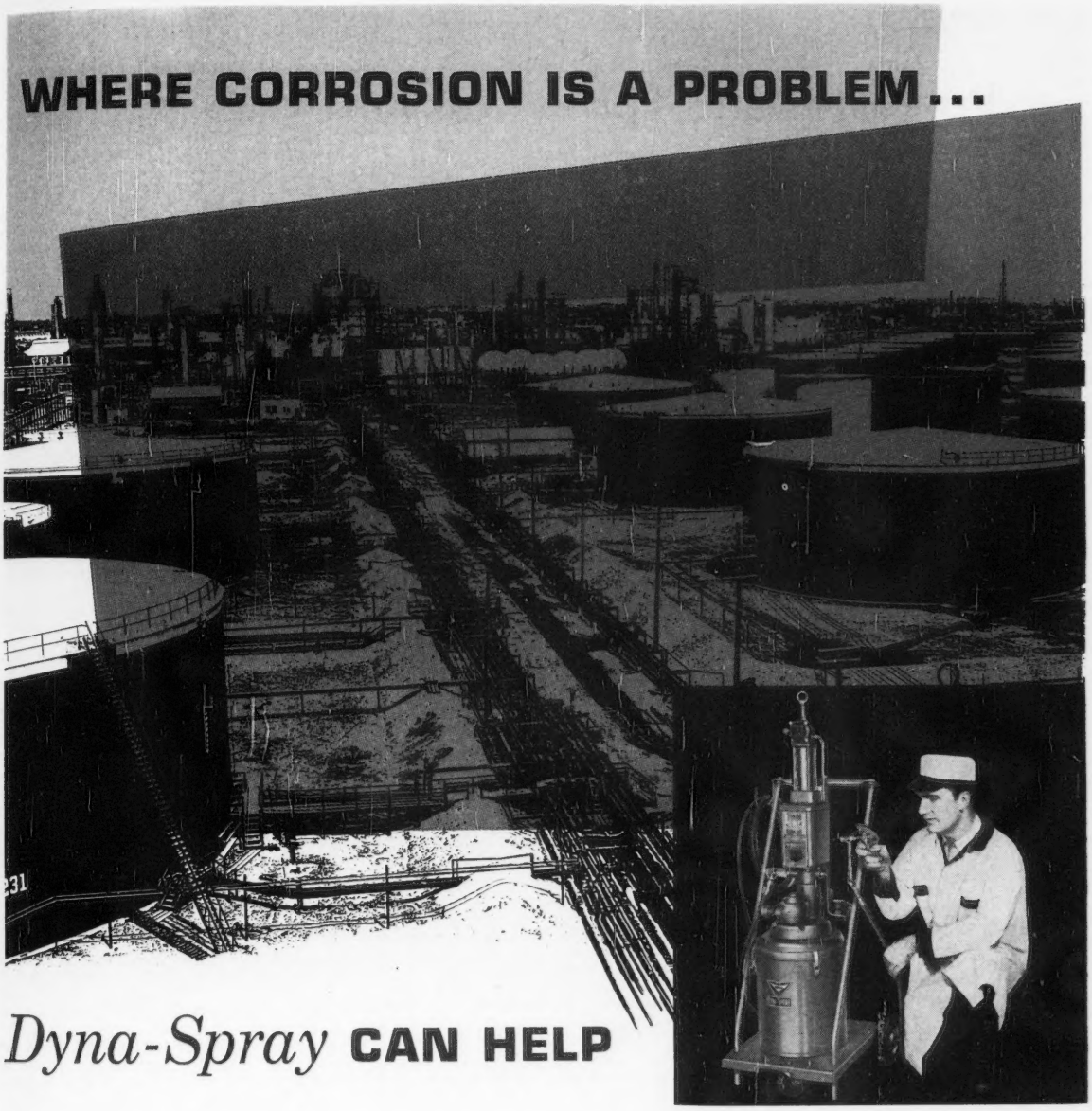
The authors wish to thank F. W. Clinard, Jr., for calling their attention to the problem and C. D. Beachem for his assistance with experiments.

The supply of doubly refined electrolytic silver by the American Platinum Works, Newark, New Jersey, is gratefully acknowledged.

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Use of

Polyester and Epoxy Resins For Chemical Plant Repairs*

Abstract

Describes procedure for using polyester and epoxy resins as repair patches for chemical plant equipment. Also discusses advantages of this type repair over conventional repairs. Case histories are given to illustrate usefulness of the resin patches. 8.8.1

Introduction

POLYESTER AND epoxy resins when cured with a selected catalyst system become hard, thermosetting plastic materials with outstanding chemical resistance. Reinforced with glass mat or glass cloth, the resins are used successfully in a variety of applications as laminated structural materials for chemical and related services. For several years an epoxy and more recently a polyester have been used as a repair or patch material for equipment in chemical service where failure occurred due to corrosion. Generally, repairs of this type are considered to be only temporary of an emergency nature. However, in many cases, they turn out to be permanent.

This article presents some experiences with the performance of resin patches for chemical plant repair, discusses their advantages and gives several case histories.

Chemical Resistance

Resins which would cure at room temperature and which would be relatively easy to use were selected for repair uses. Chemical resistance tests were made to select those with the best resistance.

Generally, these materials are resistant to non-oxidizing acids, salt solutions, water and organic acids. The polyesters are more resistant to the latter than epoxies. In oxidizing media, the resistance of both resins is variable, the polyesters being more resistant. The epoxies exhibit better resistance to polar and non-polar organic solvents than do the polyesters. In alkaline media, epoxies possess excellent resistance; polyester resistance is restricted to the lower concentration and temperature ranges. Ultimate temperature limitation of these resins depends to a great extent on formulation, but they generally can be used up to the 250-300 F range.

Surface Preparation

First requirement for successful application of polyester and epoxy repair patches is a well prepared, cleaned surface. Without the proper foundation, shrinkage during cure, although low, will result in poor adhesion with subsequent loss of bond and usefulness of the patch. Because most repairs are only temporary, minimum surface preparation should be used for economic reasons. It would be superfluous, for example, to sandblast to white metal.

Minimum surface preparation required to adequately remove rust, mill scale, grease, chemical contamination, etc., can be achieved by hand tool or power tool cleaning which usually includes a water, solvent or neutralization wash. This method does not, nor is it intended to, remove all traces of rust and scale—only that which is loose. All grease and chemical contamination should be removed before scale and rust. After cleaning, the surface must be dried. Plant maintenance men, after several applications, will be able to judge fairly well if the surface is prepared adequately. In some instances, a sweep-blast or better may be preferable and perhaps more economical than hand cleaning. The latter method, however, has proved generally to be successful.

Patching

The repair patch should be applied as soon as the surface is dry to insure that the surface does not become contaminated again. Enough resin and catalyst estimated to be sufficient to complete the repair are obtained in the proper ratio. In the field, only enough resin and catalyst are mixed which can be used in the relatively short time before cure takes place. Mixing must be thorough. Catalyst is roughly measured into resin, e.g. in a ratio of one part catalyst to four parts resin. Resins should be selected which do not require critical catalyst addition or eye dropper measurements.

An expendable brush is used to apply the catalyzed resin. One coat of resin is applied first. One or more layers of glass cloth or mat are then applied. Between each layer of cloth or mat, another resin coat is applied. Care is taken to be sure that the glass reinforcing is impregnated completely and saturated with resin and that all air bubbles are removed. This is accomplished by working the surface with the brush and rubber gloved hands. The patch is always completed with a final coat of resin. If several layers of glass mat are used, occluded air can best be removed by covering the fresh repair with a small piece of plastic fly screen and pressing or squeegeeing with a roller to remove air. The screen can then be removed and applied to another section of the repair as before. Porous starch-impregnated glass fibre can be used instead of plastic fly screen if desired.

The cloth, of course, provides the necessary support during and after cure for the resin and also the necessary tensile strength. Ten mil, 16 by 14 count, 10 oz per sq yd cloth is suitable for most jobs. For overhead work and complicated shapes, a lighter cloth is preferable: 8 to 10 mils, 18 by 18, 5 to 6 oz per sq yd. In some cases, sand, asbestos or other fillers are added to give more body to the resin so that it clings to the surface without running off. Almost any fibrous material can be used as reinforcing and almost anything as a filler.

The patch will harden rapidly, within a half hour at room temperature or even faster if the resin-catalyst ratio is adjusted properly. Final cure takes three to six hours although the repaired piece of equipment is generally put back in service within a couple of hours after completion of the repair. Heat lamps are applied to accelerate cure in some instances. The resulting patch is a dense, hard laminated solid with good chemical and mechanical properties. Finishing by grinding, sanding or painting may be desired or required at this point, but generally this is not considered necessary.

The method may appear to be more complicated and exacting than it really is. Actually, plant maintenance men became adept in the techniques after instruction and assistance on one or two jobs. After that, little supervision is required.

Advantages of Resin Patches

The main advantage of resin patch repair is that downtime for the complete job, in many instances, is only a few hours; consequently production losses are small. It is a simple inexpensive method requiring only a minimum of skilled labor. The repaired area possesses good chemical resistance and does not set-up galvanic action cells (which has happened where a more noble repair was made on a less noble piece of metallic equipment). The resins bond well to most materials and the same resins can be used for repair of all different materials.

Many times repairs can be made without shutting down equipment and in some cases right over occurring leaks if the leakage is not too great. Repairs can be made on thin-walled equipment where other repair methods such as welding would not be possible. They can be made easily on odd shaped surfaces and in relatively inaccessible areas. Repairs can also be made in areas where welding is not possible because of fire or explosion hazards. Flammable liquid and vapor need not be entirely removed from equipment when resin patches are applied. In all such cases, non-sparking tools are used to prepare the surface. Reduction in manpower needed for repairs is significant.

Case Histories

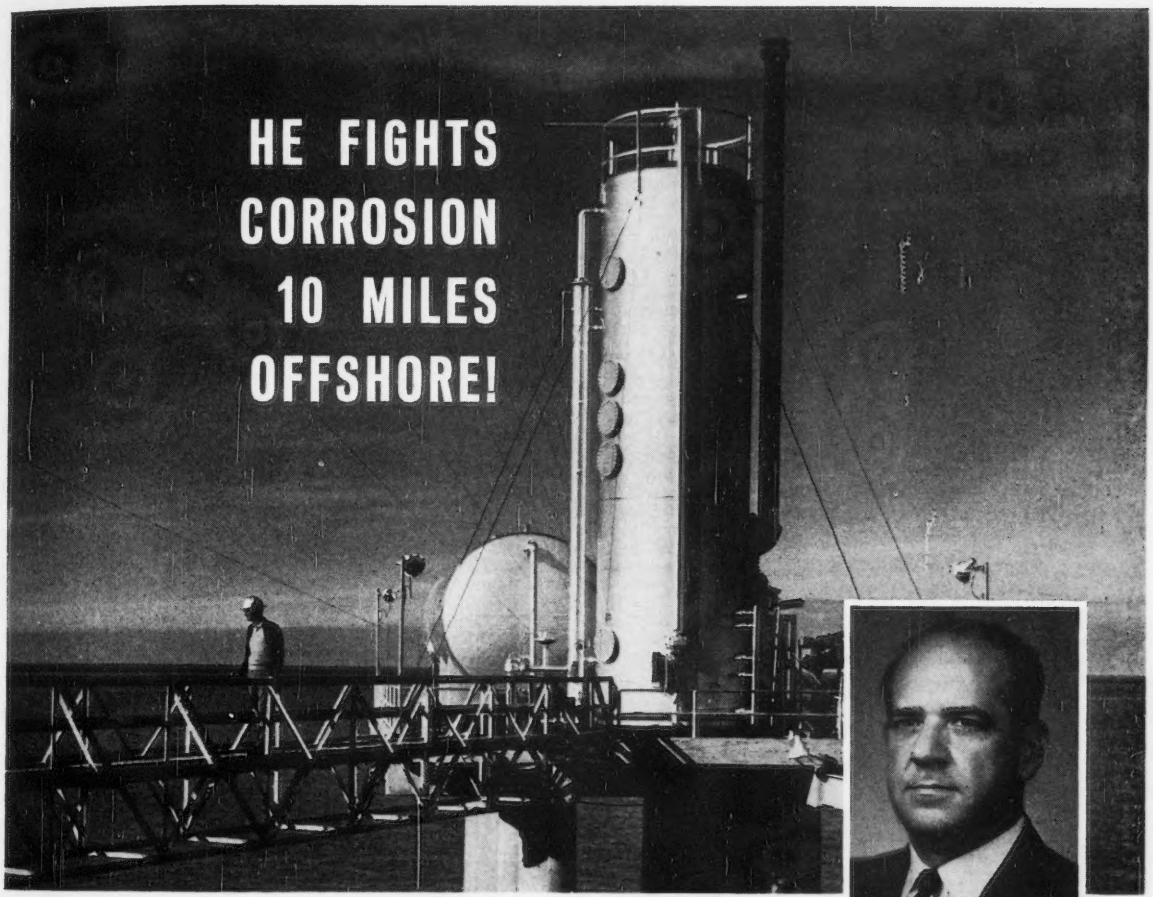
Some of the successful applications of repair patches using polyester or epoxy resin and glass cloth are illustrated by the following cases. These are listed under general headings and purposely do not mention the specific type of resin used because in most instances either resin would be suitable. Most repairs over the past several years have been made with epoxy resin. Lately, however, the trend is to change slowly to the use of polyester resin because of its lower cost and because it will do most jobs as successfully as the epoxy.

Steam Chest Repair

One of the first jobs was to repair several cracks which developed in and around nozzles on a steam chest. Cracks also developed in the main body of the shell and in several steel patches used to repair previous cracked areas. Several of the cracks were 18 inches long

(Continued on Page 24)

★ Revision of a paper titled "Use of Polyester and Epoxy Resin for Chemical Plant Repair" by Walter A. Szymanski, Hooker Chemical Corp., Niagara Falls, N. Y., presented at the North Central Region Conference, National Association of Corrosion Engineers, Cleveland, Ohio, Oct. 20-22, 1959.



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Polyester and Epoxy—

(Continued From Page 22)

and 1/16 inch wide. The chest handled slightly alkaline steam and steam condensate at or slightly above atmospheric pressure and at a temperature of about 212 F. The chest was old and was scheduled for replacement, but the replacement would not be ready for several months.

The decision was made to patch the chest with resin and glass cloth. Procedures as outlined above were followed. Repairs were completed in 6 to 8 hours by two maintenance men and one supervisor. The unit was pressure checked about 60 psi with water. Only one small leak occurred which was quickly repaired. Previously, condensate had poured out of the cracks at atmospheric pressure. The unit was back in service in about 12 hours and operated successfully for about six months when it was scrapped and the new unit installed.

The process foreman estimated that about $20,000 had been saved because the unit was back in production after such a short downtime. Previous less extensive repairs by welding required considerably more time.

The pressure that can be contained by this type of repair patch depends on several factors such as surface cleanliness, number of cloth and resin layers used, whether the cloth is wrapped completely around the piece of equipment, etc. No difficulties have been experienced in containing 60 psi using only the methods outlined above with one or two layers of cloth and no other special techniques.

Flaker Hood

A large nickel hood over a flaker was being corroded by splash of a highly concentrated organic acid salt and by its vapor and condensate. Temperatures ranged from 80 to 100 C. The colored corrosion products that formed on the hood fell into the flaker, contaminating the product. Resin patch repairs were made in place with no special surface preparation. The unit has been in operation for about six months with no evidence of failure or contamination problems.

Pipe Line

Four leaks developed in a four-inch pipe line carrying a salt solution at about 70 C and 60 psi. Resin repairs have successfully contained the liquid for almost three years.

Brick Lined Storage Tank

Leakage of concentrated mineral acid was observed on the exterior of the mild steel, acid brick lined tank. The area of failure could not be found by examination of the interior. It was considered that the brick work would have to be ripped out and replaced. Because this would be expensive, it was decided to patch the tank's complete interior with resin and glass cloth. The brick surface was sandblasted, and two layers of glass cloth and three layers of resin applied. This repair has been in successful operation at and below room temperature for about a year although not continuously.

Pointing Mortar

A sand-resin mixture (about 50 percent by weight) was used to point a section of a brick wall in an acid plant over twenty months ago. It is still in excellent condition. Previously, ordinary brick mortar had to be replaced or pointed at least once a year and sometimes every six months.

Flange on High Temperature Evaporator

Gas leaks were a problem at the gasketed surface on the flange of a direct fired high temperature evaporator. Repairs on the six-foot diameter, heavy duty flange were costly and time consuming. Resin-glass cloth repair patch was applied around the flange. The leaks were contained successfully for about 10 months even though skin temperature in this area is from 400 to 500 C. The patch charred badly in the service but did more than expected. It was originally hoped that the patch would last a week or two to provide sufficient time to fill an order.

High Pressure Patch

High pressure leaks developed around a one-inch diameter thermocouple well. Pressure ranged from 160 to 250 psi with skin temperature about 150 C. Six layers of glass cloth were used, wrapped around the well. The patch worked successfully for about two months in the continuous process when it was damaged mechanically.

Miscellaneous Repairs

Other applications of the resin patch technique include repair of water and steam lines and heating and cooling jackets on reactors. Glass lined equipment can be patched successfully by this method. Bench tops, floors, valves and pump bases have been patched. Plastic and alloy pump bodies which are badly eroded or corroded on the interior can be built up with resin filler and/or glass cloth. Pits on the outside of equipment can be filled with resin and filler. The technique of filling pits is especially useful on lined equipment where welding is not possible and where one pit may cause breakdown of an entire lining.

Summary

Repair patches using polyester or epoxy resin and glass cloth have been used successfully for several years on chemical plant equipment under many different exposure conditions. The techniques and methods involved in the application of these repair patches are relatively simple and have many advantages over more conventional methods such as welding.

Repairs of this type generally should be considered only temporary to permit operation of the equipment, without excessive loss of production, until permanent repairs can be made or until a replacement is ready for installation. Resin repairs should not be considered permanent unless other information or experience indicates that such is the case. Maintenance personnel tend to place too much confidence in this type repair, especially if they have worked on several jobs and are familiar with results. This can be dangerous if replacement or permanent repairs are not scheduled properly.

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Relationship of Microstructure and Stress Corrosion Cracking of Type 410 Stainless Steel*

Abstract

Three heats of AISI Type 410 stainless steel that exhibited different degrees of susceptibility to stress corrosion fracture in pure-water environments were investigated by electron microscopy and diffraction techniques to correlate microstructure with stress corrosion fracture behavior. In the hardened condition and tempered at 650 F, these steels cracked at stresses as low as 40,000 psi along networks of essentially untempered martensite in the prior austenite grain boundaries. Tempering at a temperature (1175 F) high enough to break up the continuous network in the prior austenite grain boundaries reduced stress corrosion susceptibility, but at the expense of a reduction in tensile properties. 3.2.2

Introduction

LABORATORY TESTS at the Bettis Plant of Westinghouse Atomic Power Department and at Knolls Atomic Power Laboratory (KAPL) of General Electric Company indicated that three heats of AISI Type 410 stainless steel exhibited different degrees of susceptibility to stress corrosion cracking in high temperature, high purity water environments. Bettis and KAPL requested assistance because the problem is important to the naval and civil-

★ Revision of a paper entitled "Relationship of Microstructure and Stress-Corrosion Fracture of AISI Type 410 Stainless Steel in Pure-Water Environments" by P. S. Trozzo and R. F. McCartney, United States Steel Corp., Monroeville, Pa.

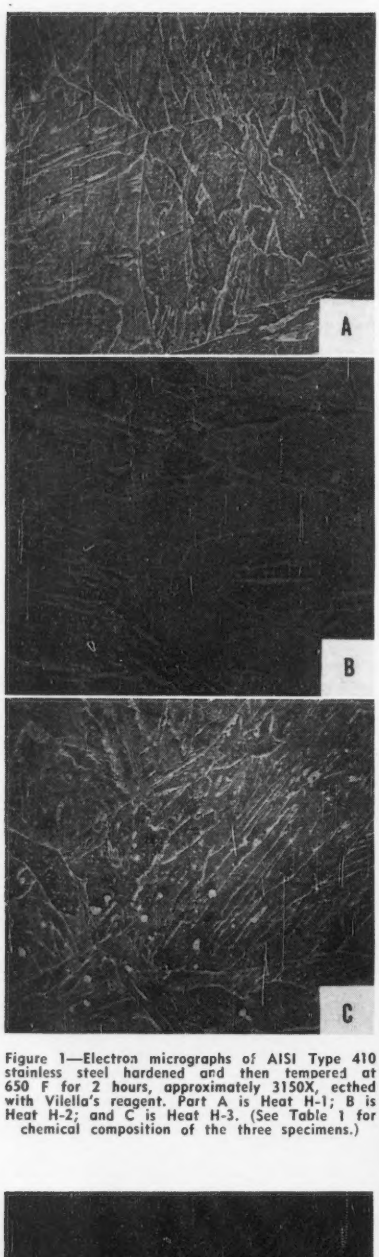


Figure 1—Electron micrographs of AISI Type 410 stainless steel hardened and then tempered at 650 F for 2 hours, approximately 3150X, ecthed with Vilella's reagent. Part A is Heat H-1; B is Heat H-2; and C is Heat H-3. (See Table 1 for chemical composition of the three specimens.)

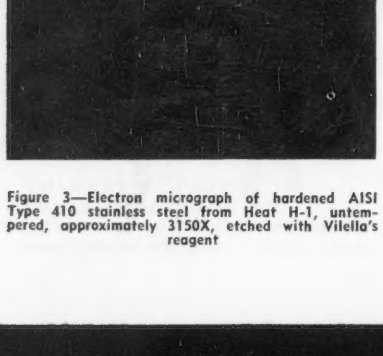


Figure 3—Electron micrograph of hardened AISI Type 410 stainless steel from Heat H-1, untempered, approximately 3150X, etched with Vilella's reagent

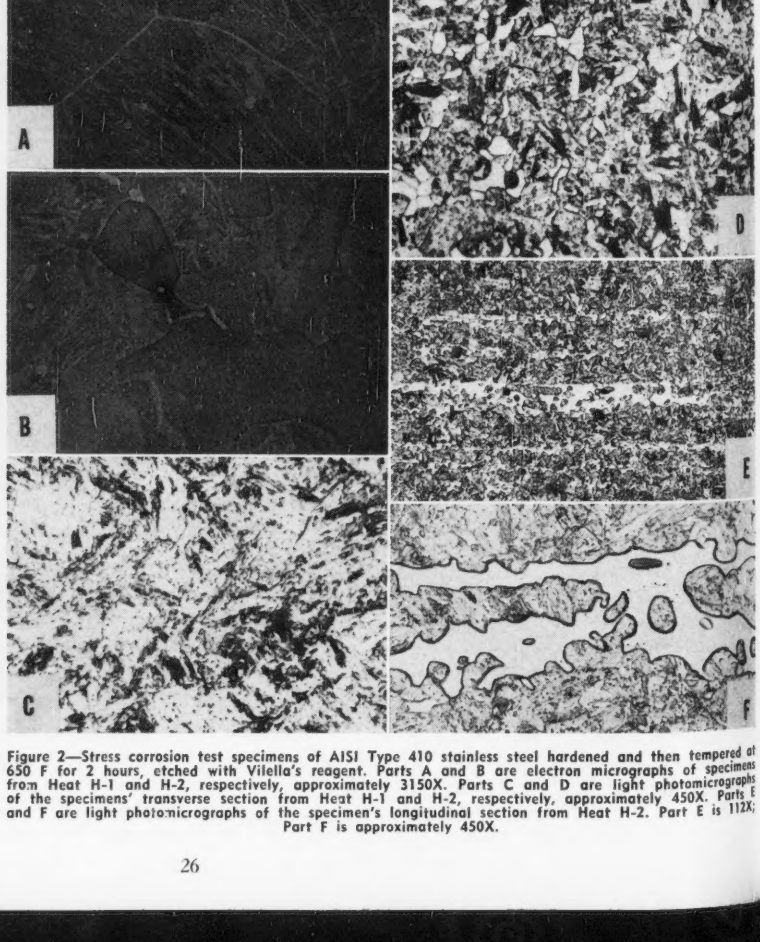


Figure 2—Stress corrosion test specimens of AISI Type 410 stainless steel hardened and then tempered at 650 F for 2 hours, etched with Vilella's reagent. Parts A and B are electron micrographs of specimens from Heat H-1 and H-2, respectively, approximately 3150X. Parts C and D are light photomicrographs of the specimens' transverse section from Heat H-1 and H-2, respectively, approximately 450X. Parts E and F are light photomicrographs of the specimen's longitudinal section from Heat H-2. Part E is 112X; Part F is approximately 450X.

ian nuclear reactor programs. The critical nature of the application of Type 410 stainless steel in the control rod drive mechanisms of pressurized-water reactors had prompted Bettis and KAPL to perform stress corrosion cracking tests in autoclaves, to determine the probability of failure in service of components constructed of Type 410 steel in the hardened condition.[1,2,3]

The laboratory tests at KAPL showed that stress corrosion failure of hardened Type 410 steel, tempered at 650 F, could occur at applied stresses of 40,000 psi in air-saturated water. There have been indications that susceptibility to stress corrosion cracking is related to the microstructure in martensitic stainless steels[4] and in austenitic steels.[5] Primary purpose of this investigation was to relate the stress corrosion cracking behavior of Type 410 stainless steel in pressurized water type nuclear reactor environments to the microstructure of the steel.

Experimental Procedure

Steels included in this investigation were from three commercial heats of Type 410 stainless steel supplied by the Bettis Plant and KAPL, who also supplied the information on chemical compositions given in Table 1. Description of the heat treatment used by Westinghouse for hardening these steels is given in Table 2. Mechanical properties of samples from these three heats in the hardened condition are shown in Table 3.

Steel samples of the three heats (H-1, H-2, and H-3) were obtained from Bettis in the annealed and in the hardened condition. Samples of H-1 and H-2 in the hardened condition were obtained from KAPL after exposure in stress corrosion cracking tests at KAPL in 300 F air-saturated water in static autoclaves, with applied stresses of 80,000 psi. Specimens from H-1 and H-3 cracked in the KAPL tests during the first week of exposure, but specimens from H-2 did not indicate any visible failure at 30X after eight weeks of exposure. Some specimens from H-1 cracked at applied stresses of 40,000 psi.

The six Bettis specimens and the two KAPL samples were examined with light and electron microscopy to determine whether the microstructures correlated with stress corrosion cracking susceptibility. As a result of the initial investigation of these samples by electron microscopy, the work was extended to include an untempered sample, samples tempered for 2 hours at 650 F plus additional tempering of 3 hours at 1175 F and samples single-tempered for 3 hours at 1175 F.

Metallographic technique used in preparing steels for electron microscopy consisted of mechanical polishing, etching with Vilella's Reagent to reveal microstructure and replication of the surface with single stage plastic films.

Discussion of Test Results

After hardening by the heat treatment described in Table 2 (tempered 2 hours at 650 F), the microstructures of Samples H-1, H-2 and H-3 were generally typical of quenched low-carbon alloy steels, as seen in Figure 1. Microstructures of H-1 and H-3, Figures 1a and 1c, are similar, but the H-2 structure (Figure 1b) differs from these in that delta ferrite is present in H-2, prior austenite grain size is smaller and aus-

(Continued on Page 28)

TABLE 1—Chemical Composition of Stainless Steel Test Specimens

Results of Chemical Analysis by the Steel Producer

	C	Mn	P	S	Si	Ni	Cr	Cu
H-1	0.12	0.49	0.017	0.015	0.48	0.28	12.38	0.10
H-2	0.10	0.40	0.014	0.026	0.33	....	12.64	....
H-3	0.15	0.39	0.014	0.008	0.30	....	12.17	....

Results of Check Analysis by a Commercial Analytical Laboratory

	C	Mn*	P	S	Si	Ni*	Cr	V	W	Al	Mo*
H-1	..	0.56	0.022	0.011	0.41	0.27	12.23	0.07	0.05	0.006	0.07
H-2	..	0.63	0.027	0.028	0.33	0.32	12.71	0.05	0.05	0.003	0.32
H-3	..	0.70	0.024	0.010	0.30	0.34	12.20	0.04	0.05	0.002	0.03

* Spectrographic Analysis.

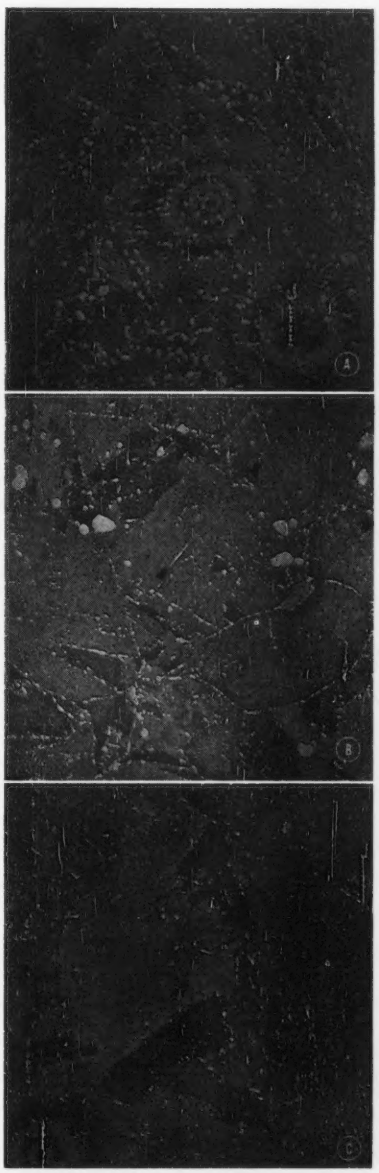


Figure 4—Electron micrographs of AISI Type 410 stainless steel, approximately 3150X, etched with Vilella's reagent. Parts A and B are from Heats H-1 and H-2, respectively, hardened and then tempered at 650 F for 2 hours and at 1175 F for 3 hours. Part C is from Heat H-1, hardened and then tempered at 1175 F for 3 hours.

TABLE 2—Heat Treatment of Samples by Bettis Plant of Westinghouse

1. Placed in furnace at 600 F.
2. Temperature increased at the rate of 400 F/hour to 1500 F.
3. Soaked at 1500 F for 1 hour.
4. Temperature increased to 1800 F rapidly and held for 20 minutes.
5. Inserted into salt bath at 1050 F—1075 F and held for 8 to 10 minutes.
6. Air-cooled to room temperature.
7. Immediately tempered for 2 hours at 650 F and air-cooled.

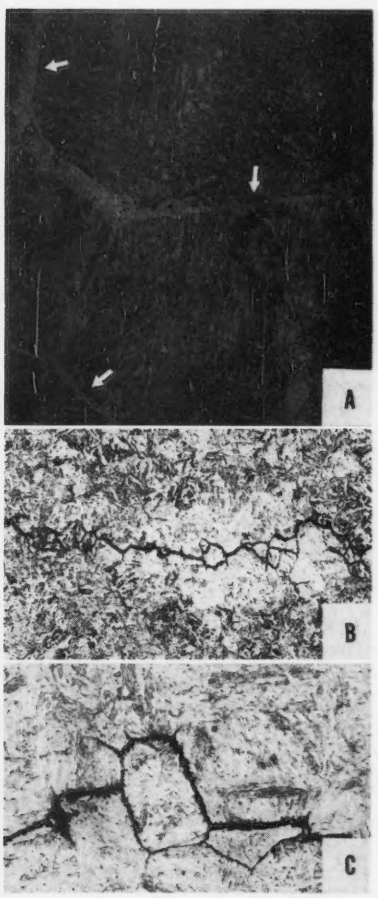


Figure 5—Cracks in longitudinal section of a stress corrosion test specimen of AISI Type 410 stainless steel from Heat H-1, hardened and then tempered at 650 F for 2 hours. Etched with Vilella's reagent. Part A is an electron micrograph, approximately 3150X. Parts B and C are light photomicrographs at approximately 112X and 450X, respectively.

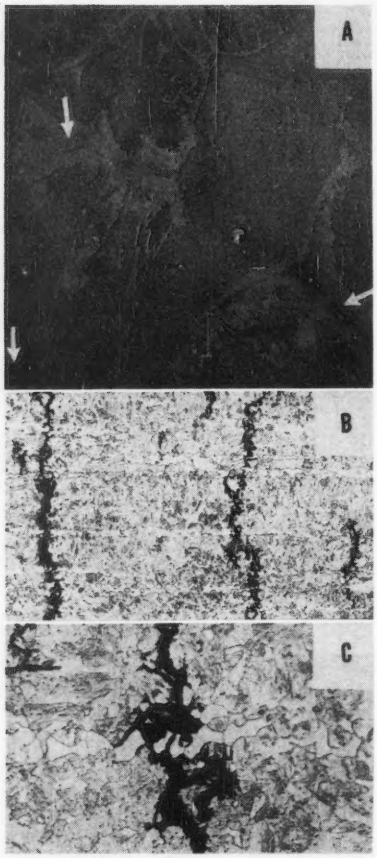


Figure 6—Cracks in longitudinal section of a stress corrosion test specimen of AISI Type 410 stainless steel from Heat H-2, hardened and then tempered at 650 F for 2 hours. Etched with Vilella's reagent. Part A is an electron micrograph, approximately 3150X. Parts B and C are light photomicrographs at approximately 112X and 450X, respectively.

Stress Corrosion—

(Continued From Page 27)

tenite grain boundaries are much less evident in H-2 than in H-1 and H-3.

Electron micrographs and light photomicrographs of stress corrosion test specimens from KAPL are shown in Figure 2. The larger austenite grains and the more prominent grain boundaries of H-1 are illustrated in the electron micrographs of H-1 and H-2 in Figures 2a and 2b, respectively. Figures 2c, 2d, 2e and 2f are light photomicrographs of H-1 and H-2. Stringers of delta ferrite are apparent in the longitudinal sections of H-2, Figures 2e and 2f; stringers of delta ferrite were not observed in H-1 and H-3.

At this stage of the investigation, the authors postulated that the unetched constituents in the prior austenite grain boundaries of H-1 and H-3 (the samples that failed in the stress corrosion cracking tests at KAPL) were higher in carbon and alloy content than the bulk composition of the steel. To investigate this phase, microstructure of a hardened (Steps 1 through 6 of Table 2) sample of steel H-1 in the untempered condition was determined (Figure 3) and was found to contain the continuous grain boundary phase also.

A reflection-electron-diffraction pattern of the untempered sample of H-1 with the grain-boundary phase etched into relief was identified as that of austenite. This proved that the grain boundary constituent was at least partly austenite. In similar diffraction analyses made on this sample after it was tempered for 2 hours at 650 F, austenite could not be detected. Amount of austenite in the untempered sample of H-1 was determined as less than the 1 percent detection limit of a quantitative X-ray diffraction method.

The presence of retained austenite and the different etching characteristics of the prior austenite grain boundaries are interpreted as evidence that the boundaries were enriched in carbon and alloy content during the quenching treatment and had higher hardenability than the matrix. Most of the boundary alloy probably transformed to martensite during quenching, but the austenite, retained in the prior austenite grain boundaries of material H-1, transformed to essentially untempered martensite during cooling from the tempering treatment at 650 F. The similarity of the microstructures of material H-1, before and after tempering, Figures 1a and 3, shows that this steel is only slightly tempered by 2 hours at 650 F. The network of untempered or perhaps slightly tempered martensite would provide an easy path for propagation of a crack and would certainly increase the susceptibility of this material to stress corrosion cracking.

On the other hand, the network of retained austenite and martensite was not as evident in steel H-2 after the hardening treatment, probably because of the smaller grain size in steel H-2 and the different transformation characteristics of a steel containing delta ferrite. The less pronounced grain boundary network, smaller prior austenite grain size and presence of delta ferrite stringers, each tending to suppress crack propagation, contributed to the superiority of steel H-2 over H-1 and H-3 in the KAPL corrosion cracking tests.

A logical remedy for the deficiency of materials H-1 and H-3 in the stress corrosion cracking tests is to temper these Type 410 stainless steels at a temperature high enough to break up the continuous grain boundary network, provided that the attendant decrease in strength can be tolerated. Microstructures of hardened H-1 and H-2 samples, after double tempering for 2 hours at 650 F plus 3 hours at 1175 F, are shown in Figures 4a and 4b. Agglomeration of carbides and absence of a grain boundary network are apparent in these micrographs.

Single tempering of steel H-1 for 3 hours at 1175 F (Figure 4c) provides a microstructure similar to that in Figure 4a. More recent stress corrosion cracking tests in autoclaves at Bettis and KAPL in reactor reference water and air-saturated water indicated that steel H-1 is not susceptible to stress corrosion cracking in autoclave tests after being tempered for 4 hours at 1150 F.

Investigation of the cracks in the specimen of steel H-1 (tempered at 650 F) exposed in the KAPL tests showed that the cracks follow prior austenite grain boundaries, shown in Figure 5.

Families of cracks were visible with the naked eye on the KAPL H-2 specimen after metallographic polishing. Electron and light micrographs of the surface of H-2 in tension in the KAPL test are shown in Figure 6. Discontinuity of the cracks at the delta ferrite-martensite boundaries is interpreted as graphic evidence of the crack-suppression effect of the delta ferrite stringers. Crack initiation probably occurred in the highly stressed network of essentially untempered martensite in the prior austenite grain boundaries.

Summary

Samples of three heats of AISI Type 410 stainless steel (H-1, H-2 and H-3) that exhibited different degrees of susceptibility to stress corrosion cracking in pure-water environments were investigated by means of electron microscopy and diffraction. This investigation was conducted to correlate the microstructure of these steels with stress corrosion cracking susceptibility in pure water media.

In the hardened condition, tempered at 650 F, steels H-1 and H-3 failed in stress corrosion cracking tests at Bettis and at KAPL, but H-2 did not fail. Microstructures of these specimens were typical of quenched low-carbon alloy steels. However, in the quenched condition, steels H-1 and H-3 contained a continuous constituent in the prior austenite grain boundaries consisting of enriched austenite and martensite. Upon tempering at 650 F, the retained austenite transformed to martensite, thus creating a grain-boundary network of essentially untempered martensite.

Cracks that resulted in failure in test specimens of steel H-1 (650 F temper) were located along these prior austenite grain boundaries. The grain-boundary network in steel H-2 was not as evident, and longitudinal stringers of delta ferrite were observed. Many small cracks were found in the test specimen of steel H-2 also, but these fissures were not continuous across the delta ferrite stringers. The less pronounced grain-boundary network, smaller prior austenite grain size and presence of delta ferrite stringers, each tending to suppress crack propagation, contributed to the superiority of steel H-2 over H-1 and H-3 in the tests.

Tempering of these steels at 1150 to 1175 F broke up the grain-boundary network in steels H-1 and H-3 and reduced the susceptibility to stress corrosion cracking in subsequent Bettis and KAPL tests.

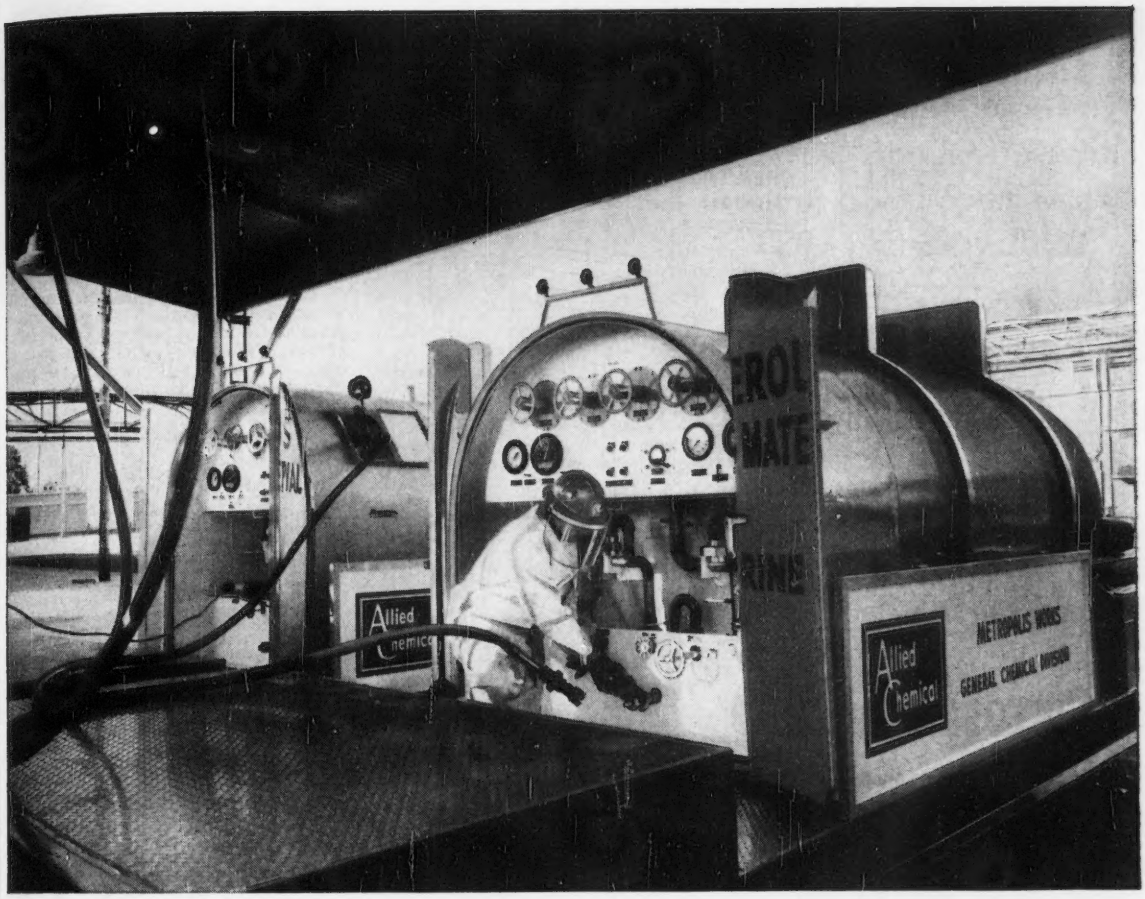
Cracks in the steels tempered at 650 F probably began in the highly stressed network of essentially untempered martensite in the prior austenite grain boundaries. However, crack propagation was suppressed in steel H-2 by its smaller

(Continued on Page 30)

TABLE 3—Mechanical Properties of Type 410 Stainless Steel Heat-Treated by Bettis Plant

Sample Identification	Tensile Strength, psi	Yield Strength, 0.2% Offset	Elongation, %	Reduction in Area, %	Hardness, R_C
H-1	192,000	140,000	9.33*	62.3	42
H-2	182,750	141,750	15.0**	60.3	39
H-3	199,300	139,160	9.33*	57.9	41

*3-inch gage length. ** 2-inch gage length.



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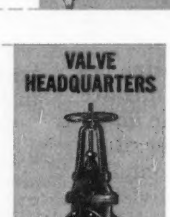
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Flexible poly pipe, ideal for water lines, drains, underground pipe or conduit. Sizes ½ to 2", long coils, NSF-approved for drinking water. *Bul. CE-57.*



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Stress Corrosion–

(Continued From Page 28)

austenite grain size, presence of delta ferrite stringers and less extensive grain-boundary network.

This type investigation, relating microstructure and stress corrosion fracture behavior, is being extended to other stainless and alloy steels at the Applied Research Laboratory.

Acknowledgments

The authors are indebted to J. R. Hunter of Bettis plant and Henry Suss of KAPL for furnishing steel samples, to A. G. Lee for the light photomicrographs, to S. C. Sekeres for metallographic assistance in electron microscopy and to the United States Steel Corporation for permission to publish this work.

References

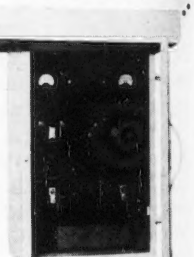
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2. E. Rau and J. V. Alger. Corrosion and Metallurgical Investigation of 1150 F Tempered Type 410 (403) Stainless Steel. WAPD-CTA (MEE) 510, June 12, 1958, Westinghouse Electric Corporation, Bettis Plant, Pittsburgh.
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4. P. Lillys and A. E. Nehrenberg. *Trans. ASM.* **48**, 327-355 (1956).
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7464 copies of NACE Technical Committee Reports published in CORROSION were sold in 1958.

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TECHNICAL COMMITTEE ACTIVITIES

Committee Officers Recently Elected

T-6 Vice Chairman

R. H. Bacon of Dow Chemical Company, Freeport, Texas, was elected vice chairman of Technical Committee T-6 on Protective Coatings. An NACE member since 1953, he is superintendent of Dow Chemical's paint department and has been in corrosion control work for almost 20 years. He will take office the last day of March.

T-4E Vice Chairman

Russel W. Lane of the Illinois State Water Survey Division, Champaign, Ill., recently was elected vice chairman of Technical Committee T-4E on Corrosion by Domestic Waters. His present position is chief inorganic chemist with the Water Survey Division. He has been a member of NACE since 1946 and has a BS from Knox College and an MS from Illinois Institute of Technology.

T-6D Officers

New Officers recently elected for Technical Committee T-6D on Industrial Maintenance Painting are Chairman F. P. Helms and Vice Chairman Donald D. Cone.



Helms

Mr. Helms is affiliated with the protective coatings department of Union Carbide Chemicals Company, Texas City, Texas. He has been an NACE member since 1955 and has a BS in chemical engineering from the University of Arkansas.

Mr. Cone, an NACE member since 1952, is associated with Texas Butadiene and Chemical Corp., Channelview, Texas. He has been in corrosion control work for over 20 years and formerly was employed by Corn Products Refining Company and Insul-Mastic Corporation of America.

These two officers will assume their T-6D office duties on the last day of March.

Greater New York Committee

The 1960 officers recently elected for the Greater New York Committee on Corrosion, affiliated with Technical Unit Committee T-7A, Northeast Region Corrosion Coordinating Committee, are Chairman W. E. Ripley of New York Central Railroad, New York City, and Vice Chairman F. E. Kulman, Consolidated Edison Company of New York, Inc., New York City.

New Jersey Corrosion Committee

Three new officers elected by the New Jersey Committee on Corrosion, affiliated with Technical Unit Committee T-7A, Northeast Region Corrosion Coordinating Committee, are as follows: Chairman R. J. Bishop of Public Service Electric & Gas Co., Newark, N.J., Vice Chairman C. W. Beggs also of Public Service Electric, and Secretary E. T. Pearson, Public Service Electric & Gas Co., Maplewood, N.J.

Columbus and Central Ohio Committee

Officers for the Columbus and Central Ohio Committee on Corrosion, affiliated with Technical Unit Committee T-7B, North Central Region Corrosion Coordinating Committee, have been elected to serve until November, 1960.

They are Chairman I. E. Davis of Ohio Fuel Gas Co., Columbus, Vice Chairman W. C. Burns, Ohio Bell Telephone Co., Columbus, and Secretary J. H. Fitzgerald, Columbia Gas System, Columbus.

Task Group T-5C-2 Has Second Meeting

The second meeting of Technical Committee T-5C-2 on Corrosion by Cooling Waters (North Central Region) will be held April 27 at the Cleveland Engineering Society Building, Cleveland, Ohio. Reservations can be made by contacting T-5C-2 Chairman William A. Hess, Standard Oil Co., Midland Bldg., Cleveland 15, Ohio.

First meeting of T-5C-2 was held October 22, 1959, in Cleveland. Discussion was held on a project for long range study by the committee and on the committee's scope of activities. The committee's scope will include any problem relative to the waterside corrosion, deterioration, fouling or scaling of all materials used in the construction, operation and maintenance of industrial equipment using cooling water common to the North Central Region.

The first meeting also included a discussion on standardization of corrosion test specimen size and evaluation of other methods of testing such as the electrical methods.

A questionnaire is being sent to interested parties in the North Central Region to learn what corrosion control systems are being used by industries, what type water is used and some of the problems being encountered.

A member described the problem of galvanic attack on the gasket surfaces of water coolers. In discussions that followed, the problem of cooling tower water deterioration was also discussed.

API-NACE Manual to Be Basis for Short Course

Plans are underway for a short course on corrosion of oil and gas well to be based on the API-NACE manual published in 1959.

The manual titled "Corrosion of Oil and Gas Well Equipment" was compiled by a special editorial subcommittee of NACE Technical Committee T-1 and was reviewed by a special subcommittee of the American Petroleum Institute.

Copies of the manual are available from the API Division of Production, 300 Corrigan Tower Bldg., Dallas 1, Texas, at $2.50 a copy.

New Anode Material For Sea Water Uses Discussed by T-2B

A new anode material for use in sea water or hot water installations was discussed at the Technical Committee T-2B (Anodes for Impressed Current) meeting held in Denver during the South Central Region Conference, October 13, 1959.

A task group member presented photographs illustrating the relative attack on high silicon iron, high silicon iron with molybdenum and the new alloy under very severe conditions. These anodes were evaluated in synthetic sea water at 200 F, one ampere per square foot in a 50-gallon drum test for 28 days.

It has been known for several years that high silicon cast iron was unsatisfactory for high temperature brackish water service. High silicon with molybdenum proved better but exhibited a tendency for premature failure due to localized pitting under certain severe conditions.

The new alloy with the same general mechanical properties of the high silicon alloys has an acceptably low and uniform corrosion rate. Preliminary results from field tests equally as severe as the laboratory tests have verified the accuracy of the observations.

Consumption rates (pounds per ampere per year) for the three anodes were as follows: high silicon iron: 18.50; high silicon iron with molybdenum: 11.80; new alloy: 0.94. The first two alloys showed severe selective attack after only seven days and continued to increase with time. This condition did not occur on the new alloy.

Increasing current density altered the attack only slightly on the new alloy. The tendency for high silicon and high silicon with molybdenum to show undercutting at taped areas seemed to be eliminated on the new alloy.

This new alloy is anticipated to have application in all salt water and high temperature fresh water services such as boiler feed water treatment units, condensate returns in refineries, heater treaters, industrial hot water tanks, pasteurizing units and many others. Persons interested in making tests with the new alloy anode can obtain anodes in standard design sizes by contacting W. T. Bryan, Duriron Company, Inc., Dayton, Ohio.

Task Group T-2B-4 Fourth Interim Report was published on pages 65t-69t in the February, 1960, issue of CORROSION.

A new task group (designated T-2B-8) was appointed to investigate and recommend materials for use in leads for impressed current anodes and for splices in such lead wires. T. J. Maitland of American Telephone and Telegraph Company, New York City, was appointed chairman of the new group.

Reports also were heard from other task groups.

Order Form

For Copies of Papers Presented at the NACE Dallas Conference, March 14-18, 1960

Use this form to order copies of NACE Conference Papers to be printed by NACE. Indicate the number of copies of each paper wanted in the appropriate place, carry the total down and compute the amount to be sent along with this order to NACE, 1061 M & M Bldg., Houston 2, Texas. Please PRINT your complete MAILING ADDRESS in the box below which will be used as a mailing label on your copies of the papers. Delivery will be as soon as possible after the conference ends.

Paper No.	TITLE	COPIES
1	Corrosion of Materials by Fluorine and Liquid Oxygen, by F. W. Fink and Earl L. White.	
4	Corrosion Detection Techniques as They Apply to Missiles in Silo Storage, by Leo E. Gatzek.	
5	Corrosion in a Large Diethanolamine Refinery Scrubbing System, by K. L. Moore.	
7	Estimating the Cost of Corrosion in Refinery Crude Units, by Norris J. Landis.	
11	Problems Arising in Corrosion Tests for Sandwich Type Construction, by J. E. Halkias.	
14	High Temperature Furnace Corrosion, by R. D. Merrick.	
15	Corrosion of Stainless Steels by Hot Hydrogen Sulfide, by F. J. Bruns.	
16	Controlling Corrosion While Chemical Cleaning, by Charles M. Loucks.	
17	Inorganic Zinc Coatings, by F. Parker Helms.	
21	Laboratory Testing of Spray Applied Lining Materials, by R. S. Foster.	
22	The How, the Why, the Cost of White Sandblasting, by Henry T. Rudolf.	
24	Application Tolerance of Protective Coatings, by M. L. Monack, J. R. Allen and F. W. Thompson.	
26	Discussion of Corrosion Problems in a Synthetic Rubber Plant, by Nick Monsour.	
27	Trowelable Coatings for Corrosion Protection, by J. H. Cogshall and A. M. Smith.	
29	Stress Corrosion Cracking in Concentrated Sodium Nitrate Solutions, by R. L. McGlasson, W. D. Greathouse and C. M. Hudgins.	
32	Galvanic Corrosion, by Thomas P. May.	
34	High Temperature Corrosion in Refinery Services, by E. N. Skinner, J. J. Moran and J. F. Mason.	
35	High Temperature Lubrication in Reactive Atmospheres, by Robert L. Johnson, Max A. Swikert and Donald H. Buckley.	
39	Discussion of the Possible Role of Autoreduction in Passivation of Iron, by E. E. Nelson.	
41	Concentration Cells and Aqueous Corrosion, by E. Schaschl and G. A. Marsh.	
44	Laboratory Investigation of Water Side Scale and Corrosion in Presence of High Process Side Temperatures, by Herman Kerst.	
	TOTAL	

Paper No.	TITLE	COPIES
46	Compositional Effects in Corrosion of Type 347 and Type 316 Stainless Steel in Chemical Environments, by C. P. Dillon.	
47	Chemical Plant Corrosion Control, by John L. Weis.	
50	Corrosion of Metals in Tropical Environments—Part 4: Wrought Iron, by C. R. Southwell, B. W. Forgeson and A. L. Alexander.	
54	Development of Thin Metal Film Corrosion Indicators, by Gilson H. Rohrback and David Roller.	
56	Use of Impressed Current Anodes in Cathodic Protection of Pipe Lines, by W. P. Noser.	
59	Problems Encountered in Cathodically Protecting Pipe Lines Crossing Railroads, by F. L. Chatten.	
60	Cathodic Protection in Desert Soils, by W. C. R. Whalley.	
61	High Temperature Corrosion Protection With Spray Metallized Coatings, by Harvey S. Miller.	
62	Ceramic Coatings for High Temperature Service, by S. W. Bradstreet.	
63	Materials Problems Associated With Uncooled Rocket Nozzles, by Jesse D. Walton and C. R. Mason, Jr.	
64	Ablation Behavior of Materials Subjected to Missile Re-Entry Heat Flux Rates, by R. M. Krupka and D. E. Taylor.	
68	Thermoplastics for Chemical Process Equipment, by Harvey E. Atkinson.	
69	Effects of Fabricating Technique on the Physical Properties of Polytetrafluorethylene and Monochlorotrifluorethylene for Corrosion and Service, by Victor G. Reiling.	
75	Inhibition of Corrosion of Commercial Aluminum in Alkaline Solutions, by J. Sundararajan and T. L. Rama Char.	
79	Plastics From the Equipment Manufacturing Standpoint, by R. McFarland.	
82	Corrosion of Carbon and Low Alloy Steels in Out-of-Pile Boiling Water Reactor Environment, by D. C. Vreeland, G. G. Gaul and W. L. Pearl.	
87	Corrosion Takes Toll on 28-Year-Old Transmission Main, by John Van Bladeren.	
92	An Investigation of the Corrosion Rate of Mild Steel in San Diego Harbor, by M. H. Peterson and L. J. Waldron.	
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(*Note: A Registrant is one who has paid the registration fee, or is registered as an exhibitor.)

Be sure to return this entire order form with your remittance. Your order must be received at NACE Central Office, 1061 M & M Bldg., Houston 2, Texas, not later than April 1, 1960. Orders received after this date cannot be filled.

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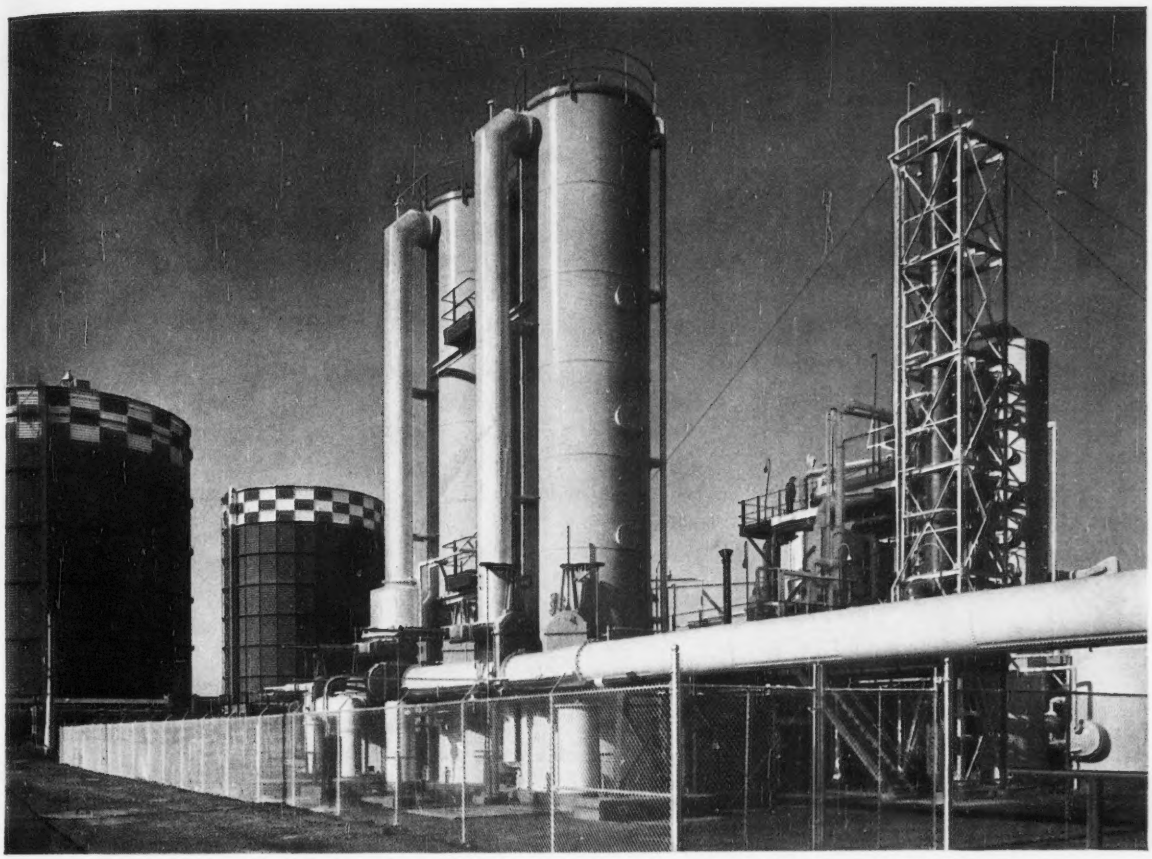
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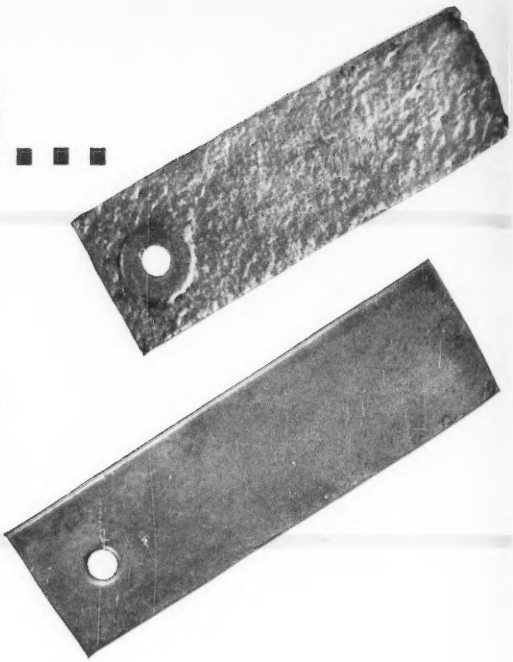
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Well	Maximum Weight Loss—Mils/year	
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** These wells receiving "anti-corrosion" treatment. Replaced by Visco.*

1 mil=.001 inch. A corrosion rate of 216 mils per year means that the corrosive attack, if uniform, would have corroded away the entire surface of the metal to a depth of .216 inches in a year. However, the real danger of high corrosion rates, as the coupon shows, is the much deeper and faster penetration occurring at localized areas.

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NACE NEWS

90 Companies to Have Exhibits At 1960 Dallas Corrosion Show

A diversity of products, items of equipment and processes in corrosion control will be on display at the 1960 Corrosion Show to be held in conjunction with the 16th Annual NACE Conference in Dallas, Texas, March 14-18. The show will feature the corrosion control products of 90 companies in 121 booths at the Dallas Municipal Auditorium.

The Corrosion Show will be open from 10 am to 5 pm on Tuesday, Wednesday and Thursday, March 15-17. No technical sessions have been scheduled for Tuesday afternoon so that conference registrants will have an opportunity to visit the show. Technical sessions and committee meetings will be held in the auditorium on the floor above the Corrosion Show.

Special Invitations Available

All exhibitors have been given the opportunity to obtain invitations from NACE for distribution to persons interested in visiting the exhibits. These invitations should be presented at the registration desk where a guest badge will be issued. About 10,000 invitations have been sent to exhibitors.

Exhibitors Added

Four companies not listed with the exhibitors on Page 44 of the January issue of CORROSION will have booths at the Corrosion Show. They are GENERAL AMERICAN TRANSPORTATION CO., Chicago, Ill., MARINDUS COMPANY, New York, N.Y., TUBE-KOTE, INC., Houston, Texas, and DOW CHEMICAL CO., MAGNESIUM DEPARTMENT, Midland, Mich.

1961 Corrosion Show in Buffalo

Information on the 1961 Corrosion Show to be held in Buffalo, N. Y., in conjunction with NACE's 17th Annual Conference, March 13-17, 1961, will be available on May 1. A mailing will be made to prospective exhibitors giving information on this show. Information also can be obtained by writing R. W. Huff, Jr., exhibition manager, NACE, 1061 M & M Bldg., Houston 2, Texas.



Bird



Smith

2 Regional Directors Will Take Office During Conference

Two new regional directors who will take office during the 16th Annual NACE Conference in Dallas are J. Dwight Bird for the Northeast Region and Arthur B. Smith for the Southeast Region.

J. Dwight Bird, an NACE member since 1947, is vice president and director of the Dampney Company, Boston, Mass. He has been in the field of corrosion control and protective coatings for thirty-one years. After his education at the University of Dallas, Texas, he was associated with General Electric Company and Ebasco. Active in NACE affairs, he has been chairman of the Boston Section and the Northeast Region.

Arthur B. Smith, an NACE member since 1949, is southeastern district manager of Amercoat Corporation, Jacksonville, Fla. Educated at the Universities of North Carolina and Colorado, he has been in corrosion control work over 15 years. Before joining Amercoat, he was associated with the U. S. Navy Bureau of Public Works, U. S. Army Engineers and the U. S. Geological Survey. He has been chairman of the Southeast Region and the Jacksonville Section.

16th Annual NACE Conference Technical Program Completed

Final changes have been made in the technical program for the 16th Annual NACE Conference to be March 14-18 in Dallas, Texas. Meetings are scheduled in the Adolphus Hotel and the Dallas Municipal Auditorium, which is 2½ blocks from the Adolphus.

Changes in Symposia Papers

One paper has been added to the High Purity Water Symposium (Thursday, March 17, 2-4:30 pm): Corrosion Study of Metals for Supercritical Pressure Power Plants, by R. C. Ulmer, Combustion Engineering, Inc., New York, N.Y.

The paper by Joseph S. Frink (Protection of New Gas Lines Bringing Natural Gas to Florida) originally scheduled in the Pipe Line General Symposium has been withdrawn.

Technical Committee Changes

Changes in the technical committee meetings have been made as follows:

The meeting of T-6K scheduled for March 14 has been cancelled.

T-6H will meet Monday, March 14, at 8:30 am in Room C of the Adolphus Hotel.

T-3D will meet Thursday, March 17, at 2 pm in Room 201 of the Municipal Auditorium.

T-5E will meet Wednesday, March 16, at 8:30 am in Room 204 of the Municipal Auditorium.

T-7 will meet Thursday, March 17, at 2 pm in Room G of the Adolphus Hotel.

The complete schedule of symposia and technical committee meetings were published on Page 32 of the January issue with changes given on Page 33 of the February issue.

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NACE's 16th Annual Conference and 1960 Corrosion Show will be held March 14-18 in the Memorial Auditorium, Dallas, Texas.

NACE Information Booth To Be at Dallas Conference

An NACE Booth will be set up in the Exhibition Hall of the Memorial Auditorium during the Dallas Conference. Open Tuesday through Thursday, March 15-17, the booth will serve as an information center for conference registrants and will have samples of all NACE literature available. Orders for copies of technical papers presented at the conference will be taken at the booth.

Southeast Region

Tidewater Section officers for 1960 are Chairman Murray S. Spicer, Chesapeake & Potomac Telephone Co., Vice Chairman Otis J. Streever, Warwick, Va., and Secretary-Treasurer John W. Berryman, Jr., District Public Works Office, Fifth Naval District, Norfolk.

•

Birmingham Section heard Donald A. Stolzman of Koppers Company, Inc., Tar Products Division, speak on protective coatings systems for steel water lines at the February 5 meeting.

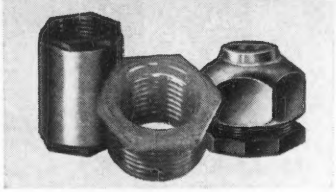
NOTICE ON PAYMENT OF 1960 NACE DUES

Invoices for 1960 membership dues have been sent to all Active and Junior NACE members. These dues were payable January 1, 1960. The NACE by-laws state that no member with dues in arrears for three months will receive the publications of the association until such dues are paid.

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Accordingly, names of Active and Junior members whose 1960 dues have not been received by March 31, 1960, will be removed from the association's mailing list.

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Western Region

Portland Short Course To Include Fundamentals

Plans are being completed for the Portland Section Short Course on Corrosion Fundamentals to be held April 27-29 in the Public Service Building Auditorium.

The first course of its type to be held in Oregon, the short course will consist of basic training in corrosion theory, corrosive environments, recognition of corrosion, control and detective measures and corrosion prevention.

Registration fee for the course will be $15. Advance registration can be made through Curtis Lund, Portland Section, P. O. Box 2426, Portland 14, Oregon.

The first day will be devoted to two-hour lectures on fundamentals of corrosion, recognition of corrosion and instrumentation to measure and evaluate the extent of corrosion damage.

The second day, April 28, will be devoted to lectures on corrosion control methods and evaluation of protective coatings. Time is also being planned for discussion of literature sources in the field of corrosion.

The last day of the course will consist of two-hour lectures on cathodic protection, various corrosive environments and corrosion prevention.

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Western Region's 1960 Conference will be Oct. 6-7 at the Sheraton Palace Hotel in San Francisco, Cal.

Los Angeles Section heard a talk on plastic coatings in West Texas Oil fields by Burtell Martin of Ferro-Martin and Harry Kipps of Signal Oil and Gas at the January 20 meeting. This was a report on the Permian Basin Corrosion Tour, sponsored by the Permian Basin Section in Texas. Fifty-six members and guests were present at the section meeting.

•

San Francisco Bay Area Section had a slide-talk on surface preparation by sandblasting at the February 9 meeting. The presentation was made by John P. Dempsey of Clementina, Ltd., from a paper by E. W. Oakes of Houston, Texas.

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Portland Section officers for 1960 are Chairman John Van Bladeren, Northwest Natural Gas Co., Vice Chairman George K. Merz, Portland General Electric Co., and Secretary-Treasurer Keene R. Shogren, Northwest Natural Gas Co.

•

Portland Section's Corrosion Control Short Course will be held April 27-29.

North Central Region

Chicago Section, for its January 19 meeting, heard R. J. Hafsten of Standard Oil of Indiana speak on combatting refinery corrosion.

The first woman to join the section was present. She is Miss Barbara Northan of Nalco Chemical Company.

Committee assignments made recently are as follows: Richard Rue of Insulmastic Inc., program; Harry Anderson of Swift & Company, membership; and Robert Weisert of Sales Engineering Inc., publicity.

•

Detroit Section had 21 members and 18 guests at the January 21 meeting. A "Tank Lining Forum" was the technical program. Speakers were W. G. Cathcart, Tank Lining, Inc., L. Woerner, Automotive Rubber Co., and H. M. Coleman, General American Transportation Co.

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Southwestern Ohio Section heard Robert Pierce of Pennsalt Chemical Corporation speak on corrosion resistance masonry construction at the January 26 meeting.

•

Greater St. Louis Section has scheduled a talk on cooling water corrosion by Ray Baum of Hagen Chemical and Controls, Inc., for the March 7 meeting.

Bernard Husock of Harco Corporation, Cleveland, Ohio, was guest speaker at the February 8 meeting. His topic was practical aspects of cathodic protection.

F. H. Langenfeld of Monsanto Chemical Company spoke on synthetic fluids in the Space Age at the January 14 meeting.

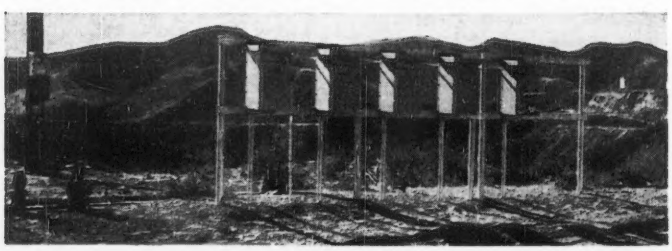
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The 1960 North Central Region Conference will be held Oct. 19-20 at the Schroeder Hotel in Milwaukee, Wis.

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226,800 copies of 25 NACE Technical Committee Reports were distributed in 1957.

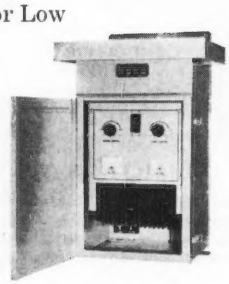
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Advantages:

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Manufacturers of AC to DC Equipment Since 1932



A ONE-COAT SYSTEM!

New PRUFCOAT PRIMASTIC gives you these extraordinary benefits...

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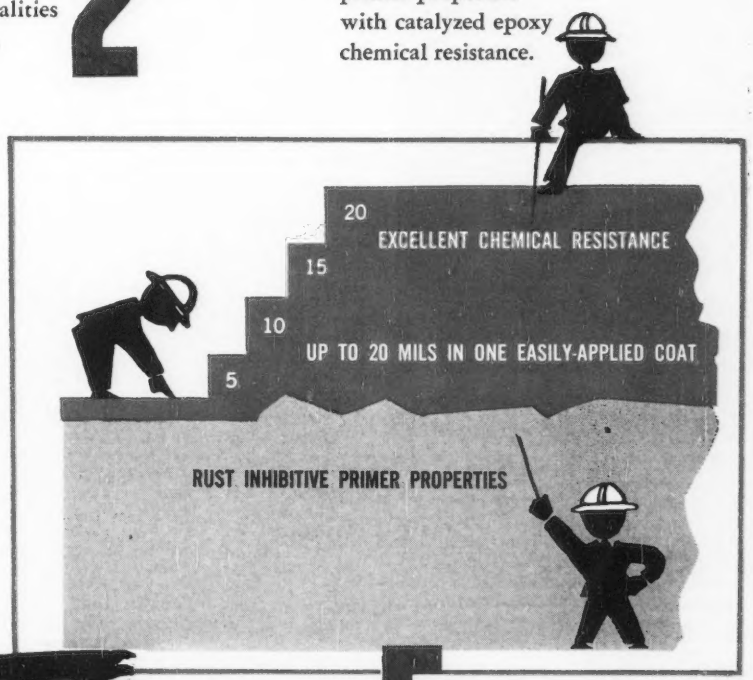
Primastic uniquely combines rust-inhibitive primer properties with catalyzed epoxy chemical resistance.

2 APPLICATION LABOR COSTS CUT 50% OR MORE

Just one brush or spray coat gives you the thickness and corrosion-control qualities of multiple-coat systems.

3 LOW MATERIAL COST PER MIL FOOT

Because of Primastic's high total solids and moderate price per gallon, cost per mil foot barely exceeds one cent.



4 MAINTENANCE COSTS SLASHED

Primastic alone can solve most of your corrosion problems —even those involving *severe* inorganic and organic chemical exposures.

5 SIMPLE, SAFE APPLICATION

No capital investment in special equipment required.
No special application skill needed.
Flash point exceeds 100°F.

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HOUSTON SECTION SHORT COURSE held January 28-29 at the Rice Hotel had a total registration of 295—an increase of 218 percent over the 1959 short course. Photographs above taken during the two-day course show (Figure A) some registrants looking over one of the cathodic protection demonstrations. Marion E. Frank, of Tennessee Gas Pipe Line Co., chairman of the session on transmission pipe lines systems, is shown in Figure B discussing his cathodic protection demonstration to some of the registrants. Maurice A. Riordan of Rio Engineering Co., Houston, general chairman of the short course, is shown in Figure C with one of the experiment apparatus used in a lecture.

ASA Suggests Standards For Shop Fabricated Tanks

A general conference of 25 representatives of the chemical industry, tank manufacturers, corrosion engineers and metal suppliers met at headquarters of the American Standards Association in December to recommend initiation of a standardization project for shop fabricated metallic storage and processing tanks used by the chemical industry.

Among the features to be standardized are capacities, dimensions and physical properties including corrosion resistance.

Walter H. Burton of the General Chemical Division of American Chemical Corporation, Camden, N. J., is the NACE representative to ASA.

South Central Region

Houston Section Has Enrollment of 295 At '60 Short Course

Registration jumped 218 percent at the 1960 Houston Section's two-day course on Practical Corrosion Control Systems over the section's 1959 short course. Total registration for the January 28-29 course held at the Rice Hotel was 295. Registration for the 1959 short course was 135.

Registrants represented 13 states including Texas and neighboring states, Mississippi, Illinois, Alabama, California, Kentucky, Colorado and Florida.

Industries represented by the registrants included oil and gas production, refining, metals, coatings and paints, municipal housing agencies, state institution maintenance, pipe lines, laboratory personnel, architects, consulting engineers, petrochemicals, utilities, construction contractors and sales personnel from all industries related to corrosion control.

The two-day course was scheduled so that the second day's activities were divided into five areas of interest with concurrent sessions. The five areas were (1) transmission pipe lines systems, (2) oil and gas production, (3) refineries, chemical, petrochemical and industrial plants, (4) utilities and (5) commercial and residential design—heating and air conditioning. Seven sessions for each of the five areas were held so that persons in a given interest area could hear speakers giving information more directly related to a specific industry rather than on a more general area.

Replies on a special evaluation sheet given to each registrant indicate that the new scheduling arrangement for the short course was helpful and that the course was well received.

Each of the five interest area sessions on the second day ended with round table discussions.

General chairman for the short course was M. A. Riordan, Rio Engineering, Houston. Vice chairman was Marion E. Frank of Tennessee Gas Pipe Line Company. Other committee members were Treasurer and Registrar Ben Russell, Rio Engineering, T. M. Heitman of United Gas Corporation in charge of arrangements and Rudy Zorn of Cameron Iron Works in charge of publicity.

•

North Texas Section had Frank L. LaQue, ASTM President and vice president and manager of development and research at International Nickel Company, as guest speaker at the February 11 meeting. His subject was "What We Know and Don't Know About Corrosion."

•

Alamo Section heard Harry Aschan of Good-All Electric speak on electric rectifiers at the January 19 meeting.

•

Shreveport Section had A. W. Peabody of Ebasco Services, Inc., as guest speaker at the February 2 meeting. He spoke on the cause and solution of pipeline corrosion interference problems.

•

East Texas Section had Phil Brock of Dowell, Inc., Lafayette, La., speak on inhibitor squeeze at the January 26 meeting.

•

Corpus Christi Section showed the two-part film "Soil Corrosion of Pipelines" and "Coating Systems" at its January 26 meeting. Moderator was Ray Sims of the City Gas Division.

•

Sabine-Neches Section meeting for January 28 included a paper titled "Some Corrosion Problems in Water Wells." The paper was presented by K. E. Moerhl, director of research, Layne Research Division of Layne and Bowler, Inc.

•

Permian Basin Section had R. L. McGlasson of Continental Oil Co., Ponca City, Okla., as guest speaker at its January 12 meeting. His topic was "Getting the Most From Your Sucker Rod Dollar."

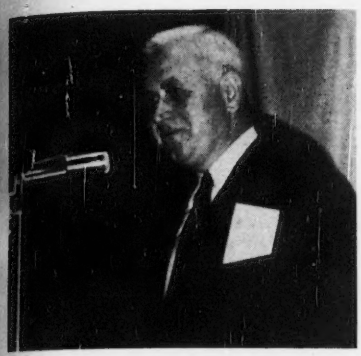
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Central Oklahoma Section, for its January 11 meeting, heard a paper presented by Bob Nee on pipeline coating for underground use.

Officers for the section recently elected are Chairman Frank Burns, Vice Chairman Truel Adams and Secretary-Treasurer John A. Knox.

•

The 1960 South Central Region Conference will be held October 25-28 at the Mayo Hotel, Tulsa, Okla.



F. L. LaQue, International Nickel Co., Inc., told a joint ASTM-NACE meeting February 9 at Houston there is a good future in corrosion control work because it probably will always be an occupation for specialists.

Kuhn Receives 1960 Medal From Louisiana Society

Robert J. Kuhn, an NACE member and consulting engineer in New Orleans, was awarded the Louisiana Engineering Society's Technological Accomplishment Medal for 1960 at the society's annual meeting January 7. The medal was presented by James M. Robert, dean emeritus of Tulane University's School of Engineering.

Mr. Kuhn received the NACE Frank Newman Speller Award in corrosion engineering in 1958.

His experience in corrosion control began in 1923 after he graduated from Tulane University with a degree in mechanical and electrical engineering. He was electrolysis engineer for New Orleans Public Service Company until 1936, when he entered the consulting business.



Kuhn

He has written over 40 technical papers for presentation before national societies and conferences.

Northeast Region

Baltimore-Washington Section scheduled Nathan W. Morgan, bridge engineer for the Bureau of Public Roads to speak on corrosion problems in highway maintenance for the section's February 16 meeting.

•

Pittsburgh Section, at its February 4 meeting, had Robert J. Schmitt of United States Steel Corporation speak on the role of corrosion testing in the coal-chemical plant.

•

Greater Boston Section saw a slide presentation on corrosion control in the Middle East at its January 13 meeting. The presentation was made by Leon P. Sudrabin of Electro Rust-Proofing Corporation. Attendance was 27 members and 15 guests.

Corrosion Reaction Variables Are Big Challenge, LaQue Says

Anomalies that crop up in corrosion control testing, largely as a result of the multiplicity of variables in most corrosion reactions are a formidable challenge. This observation and other remarks that ranged over a wide spectrum of corrosion problems, was made by F. L. LaQue, vice president of International Nickel Co., Inc., New York, before the annual Southwest district meeting of American Society for Testing Materials and NACE Houston Section February 9 at the Houston Engineering and Scientific Society Building. He is president of ASTM and a former president of NACE.

Drawing from his own extensive experience and from the voluminous data collected by his company, Mr. LaQue posed some anomalies connected with corrosion testing. These included a report on tests which indicate that success achieved by protecting cathodically surfaces subjected to cavitation damage is the result of the cushioning effect of hydrogen formed on the metal surfaces, rather than on any limit on the corrosion of the material by the current used.

Interest Is in Speed Only

Many corrosion researchers seem to be interested in speed rather than accuracy in many of their data, Mr. LaQue observed. This often leads to erroneous conclusions and to costly mistakes. In making cavitation tests, for example, reliance on laboratory magnetostriction equipment to produce reliable data on control measures often is erroneous.

In the atmospheric corrosion protection field, for example, Mr. LaQue observed, tests are loaded on the side of mechanical properties of materials.

Tests now under way by ASTM to help determine the relative corrosivity of atmospheres have produced some unexpected results. The difference in the relative corrosivity of environments now is found to be on a ratio of 2000 to 1 as demonstrated by differences in effect on specimens exposed in a Canadian location compared to others at Kure Beach.

Exact Determination Difficult

Exact determination of the effect of atmospheric corrosives is difficult, he said. It is a well known fact that the weather prevailing at the time test specimens are first exposed can have a marked effect on the corrosion rate.

So far, Mr. LaQue said, there is no reliable way to measure any characteristic of the atmosphere quantitatively to permit an accurate estimate of its corrosivity.

About 75 persons were present for the dinner meeting.

The April meeting of Houston Section will be the annual "Student Night" when students from engineering schools in the vicinity will be guests of the section. The program will consist of a panel of corrosion engineers who will discuss "Corrosion Control Work as a Future."

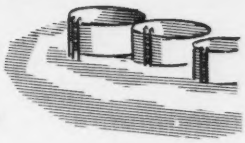
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7464 copies of NACE Technical Committee Reports published in CORROSION were sold in 1958.

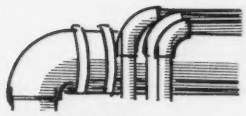


PROBLEMS IN . . .

TANKS AND TANK TRAILERS?



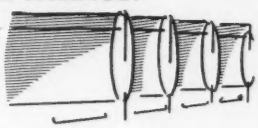
PROCESS PIPING?



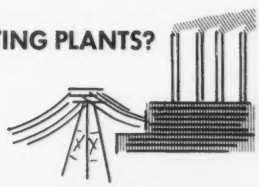
STEEL AND CONCRETE IN PROCESS AREAS?



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NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1960

March 14-18—16th Annual Conference and 1960 Corrosion Show. Dallas, Texas, Memorial Auditorium.

Oct. 6-7—10th Annual Western Region Conference, Sheraton-Palace Hotel, San Francisco, Cal.

Oct. 6-8—Southeast Region Conference, Dinkler-Plaza Hotel, Atlanta, Ga.

Oct. 11-14—Northeast Region Conference. Prichard Hotel, Huntington, W. Va.

Oct. 19-20—North Central Region Conference. Schroeder Hotel, Milwaukee.

Oct. 25-28—South Central Region Conference, Mayo Hotel, Tulsa.

1961

March 13-17—17th Annual Conference and 1961 Corrosion Show, Buffalo, N. Y., Hotel Statler.

Oct. 4-6—Western Region Conference, Hotel Multnomah, Portland, Oregon.

Oct. 9-11—North Central Region Conference, St. Louis, Chase Park Plaza Hotel.

Oct. 24-27—South Central Region Conference, Houston, Shamrock Hotel.

Oct. 30-Nov. 2—Northeast Region Conference, New York City, Hotel Statler.

Southeast Region Conference, Miami, Fla., in conjunction with Miami Section's short course.

1962

March 19-23—18th Annual Conference and 1962 Corrosion Show. Kansas City, Municipal Auditorium.

October 9-11—North Central Region Conference.

October 16-19—South Central Region Conference, Hilton Hotel, San Antonio, Texas.

Southeast Region Conference, Birmingham, Ala.

Western Region Conference. San Diego, Cal.

SHORT COURSES

1960

April 27-29—Portland Section Corrosion Control Short Course.

May 11-13—Niagara Frontier Section Symposium on Organic Coatings. Hotel Niagara, Niagara Falls, N. Y.

June 1-3—5th Annual Appalachian Underground Corrosion Short Course, West Virginia University, Morgantown, W. Va.

September 28-30—Central Oklahoma Section 1960 Corrosion Control Short Course, at University of Oklahoma, Norman.

October 3-5—Corrosion Control Short Course, sponsored by Western Region and University of California.

•

In 1958, there were over 11 million privately and publicly owned trucks on U. S. streets and highways, representing 17 percent of the total U. S. motor vehicle registrations.

•

News intended for publication in CORROSION should be in Houston no later than the 10th of the month preceding month of publication.

Color Slide-Talk on 1960 Corrosion Show

A color slide illustrated talk covering many of the exhibits at the 1960 Corrosion Show will be prepared for use by NACE's sections. R. W. Huff, Jr., NACE Exhibits Manager, said color photographs will be taken of booths of those exhibitors that have shown an interest. Each exhibitor will have the opportunity also to include a photograph of one product, together with a short commentary.

Because of the wide interest in the Corrosion Show and because many NACE members will not be able to see the show in Dallas, Mr. Huff reasoned that many sections will want to present the slide talk at a meeting. It is expected that the presentation will consist of about 100 slides, with a prepared commentary concerning the equipment in the photographs. Section program chairmen who would like to schedule the slide show are invited to write G. A. Rolak at NACE Central Office for a booking date.

The show will be available for use shortly after April 1. Three copies will be prepared.

Vinyl Insulation Used

Icy sidewalks are eliminated at the Eastwood Shopping Center, Pittsburgh, Pa., by vinyl insulated heating wires installed just below the surface of the walks.

Insulated with rigid polyvinylchloride for protection from abrasion and corrosion, the heating wire will keep ice melted and eliminate the need for salt on sidewalks.

Scrap Plastics Not Safe

Scrap plastic used in making pipes for handling drinking water presents a hazard to public health in the opinion of Walter B. Tiedeman, executive director of National Sanitation Foundation Testing Laboratory. Some pipe materials include toxic substances readily extractable by slightly aggressive, potable waters, it was said.

DEATHS

Harold D. Williams, an NACE member since 1953, died January 17. He was an inspector and port engineer with the Keystone Shipping Co., Long Beach, Cal. He supervised installation and maintenance of corrosion control systems on oil tank vessels.

•

Jack R. Woodard of Phillips Petroleum Co., Bartlesville, Okla., died December 31. An NACE member since 1957, he had a BS in mechanical engineering from Oklahoma A & M College. He was corrosion engineer with Phillips Petroleum's production department.

Two More Sources Acquired for NACE Corrosion Abstracts

Two more sources of abstracts of corrosion literature have been acquired by NACE. The U. S. Department of Commerce, Office of Technical Services has authorized the association to use abstracts from its periodical TECHNICAL TRANSLATIONS and also from the U. S. Government abstract journal, U. S. GOVERNMENT RESEARCH REPORTS.

The translations journal lists translations of foreign language technical information while the government monthly periodical abstracts reports on research work done by or by contract for the federal government and its agencies.

It is the intention of the NACE to select abstracts referring to literature which has corrosion control interest and include them in the association's Abstract Punch Card Service, Bibliographic Surveys of Corrosion Control and in the Corrosion Abstracts section of CORROSION.

NACE has had access to some of the reports covered by these two publications in releases issued by OTS. This new agreement, however, assures a better chance to get complete coverage of the two areas represented. Abstracts taken from the translations journal will be coded "TT" and from the government research journal "GRR."

The addition of these two sources brings to 24 the number of agencies authorizing NACE to use their abstracts.

Copies of Corrosion Needed for Libraries

Copies of the January, February, March and September 1959 issue of CORROSION are needed to complete Volume 15 for library subscribers.

Any CORROSION reader who no longer needs to keep those issues for reference is asked to send them to the Central Office, NACE, 1061 M & M Bldg., Houston 2, Texas, attention N. E. Hamner. Issues received will be sent to library subscribers.

•

Lapel pins approximately 7/16 inches high, made of gold with inlaid red enamel and a ruby center, are available to NACE members only. Price is $10. Address orders to Executive Secretary, 1061 M & M Bldg., Houston 2, Texas.

Slide-Talk Available On Surface Preparation

A color slide presentation on metal surface preparation by sandblasting is available on loan from NACE Central Office. The talk is based on a paper by E. W. Oakes, Clementina, Ltd., Houston, presented at the NACE Annual Conference in Chicago, 1959. The illustrations used are 35mm color slides.

Requests should be sent to Gilbert Rolak, 1061 M & M Bldg., Houston 2, Texas.



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Just as the check of a good watermelon is the plug, the proof of the puddin' at TLC is the test disc from the actual run of the customer's pipe.

At TLC, you can be sure your tubing is lined flawlessly because during the cycling of your 172 joints on TLC's "Autotronic®" lining unit, (one oven load carries 5000 ft.), checks are made periodically on discs imbedded into a test joint. These discs are checked during the multiple lining and multiple baking cycles. Checks are made on continuity of film, character of film, thickness of film, and cure of film. One of the discs, after completion of the run, is sent to the customer for inspection, checking and approval. At TLC "Autotronic®" and "Thermo Pickling®" assure highest quality.

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Canadian Region

Over 300 Registered For Eastern Division Conference

Over 300 registered for the Canadian Region's Eastern Division Conference held January 18-21 in Toronto. Twenty-seven technical papers were presented in five symposia on various phases of corrosion control problems in the pipe line, refining and chemical process industries.

•

Calgary Section officers for 1960 are Trustee G. G. Mainland, Imperial Oil Co., Ltd., Chairman S. N. Gell, Canadian Western Natural Gas Co., Ltd., Vice Chairman J. S. Watson, Government of Alberta, Secretary-Treasurer F. S. Fobes, Pi-Pro Products Ltd., and Membership Chairman Albert Dinnigan, British American Paint Co., Ltd. Program chairman is Vince Milo of Shell Oil Company of Canada, Ltd.

•

Edmonton Section had Glen Mainland of Imperial Oil, Ltd., Calgary, as its guest speaker at the January 28 meeting. His topic was functions and operation of a corrosion research laboratory.



Thousands of miles of underground pipe feed natural gas to factories . . . other pipelines distribute oil and other petroleum products. And every foot . . . every inch . . . could corrode and leak anytime, day or night.

Electro Rust-Proofing can assist in the big job of corrosion control. Assist with surveys, design recommendations and actual installation of corrosion control systems.

Even if you have your own corrosion engineering group, ERP can swing extra manpower and engineering experience onto rush jobs—supplement your own team on the special problems.

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SECTION CALENDAR

March

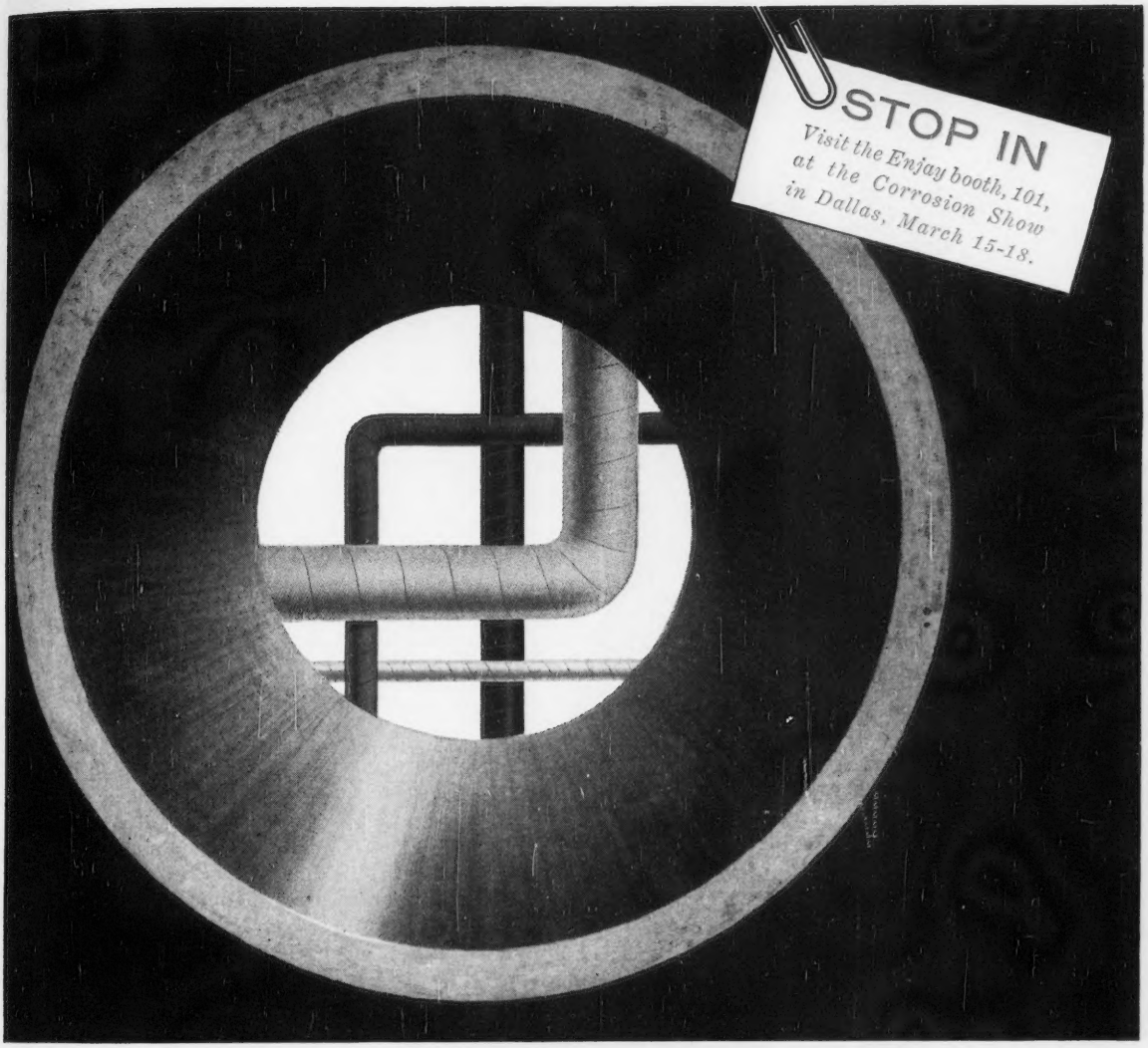
1 Shreveport Section. Production equipment. Capt. Shreve Hotel.
3 West Kansas Section.
3 Pittsburgh Section. Design for Corrosion Prevention in the Process Industry, by F. L. Whitney, Jr., Monsanto Chemical Co.
7 North Texas Section.
8 San Francisco Bay Area Section.
8 Montreal Section. Inhibitors.
9 Greater Boston Section. "What We Know and Don't Know About Corrosion," by Frank LaQue, International Nickel.
15 Chicago Section. Lead Sheath Cable.
23 Los Angeles Section.
24 Edmonton Section.
24 Detroit Section. Annual Paint Meeting. R. Gackenbach of American Cyanamid Co., guest speaker.
28 Tulsa Section.
29 Panhandle Section.
29 Southwestern Ohio Section. John Frank, National Distributing Corp., guest speaker. To be in Cincinnati.
31 Sabine-Neches Section.

April

4 North Texas Section.
5 Shreveport Section. Capt. Shreve Hotel. Review of 1960 NACE Conference.
7 West Kansas Section.
7 Pittsburgh Section. Ladies' Night.
12 San Francisco Bay Area Section.
19 Chicago Section. Drainage Problems.
19 Baltimore-Washington Section. Corrosion Preventive Measures Necessary in Design and Construction of an Underground Pipeline, by E. C. Hine and R. E. Barrans.
21 Southwestern Ohio Section. Evaluation of Coatings for Protection of Steel at Elevated Temperature, by Edward Norris of Metcut Research Assoc., Inc., to be held at Connersville, Ind.
25 Tulsa Section.
26 Panhandle Section.
26 Detroit Section. Meeting concurrent with ASTM. Speaker to be Frank LaQue of International Nickel Co., Inc.
28 Edmonton Section.
28 Sabine-Neches Section.

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News Deadline for CORROSION is the 10th of each month.



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Enjay Butyl rubber, combined with plastics, forms wrapping tapes which are unexcelled for pipeline protection.

This laminated tape consists of a layer of tough, resilient Butyl bonded to plastic film. Applied directly or over a Butyl based primer, the wrapping bonds permanently to the pipe. Results? Absolutely *no* moisture penetration. Impermeable to gases. Outstanding resistance to soil acids and other destructive corrosives. Good dielectric character combined with a high degree of impact and abrasion resistance.

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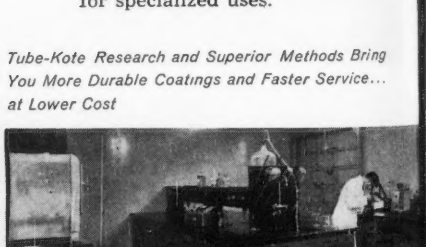
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When you specify Tube-Kote, you have every right to expect a far superior coating.

Tube-Kote's outstanding success in preventing corrosion is a result of constant and careful attention to many details. Ingredients must be blended within minute tolerances. Preparation of the metal must be carried on under strict supervision and inspection. Application must be made under near perfect conditions, and curing must be precise to meet the harsh requirements of production use.

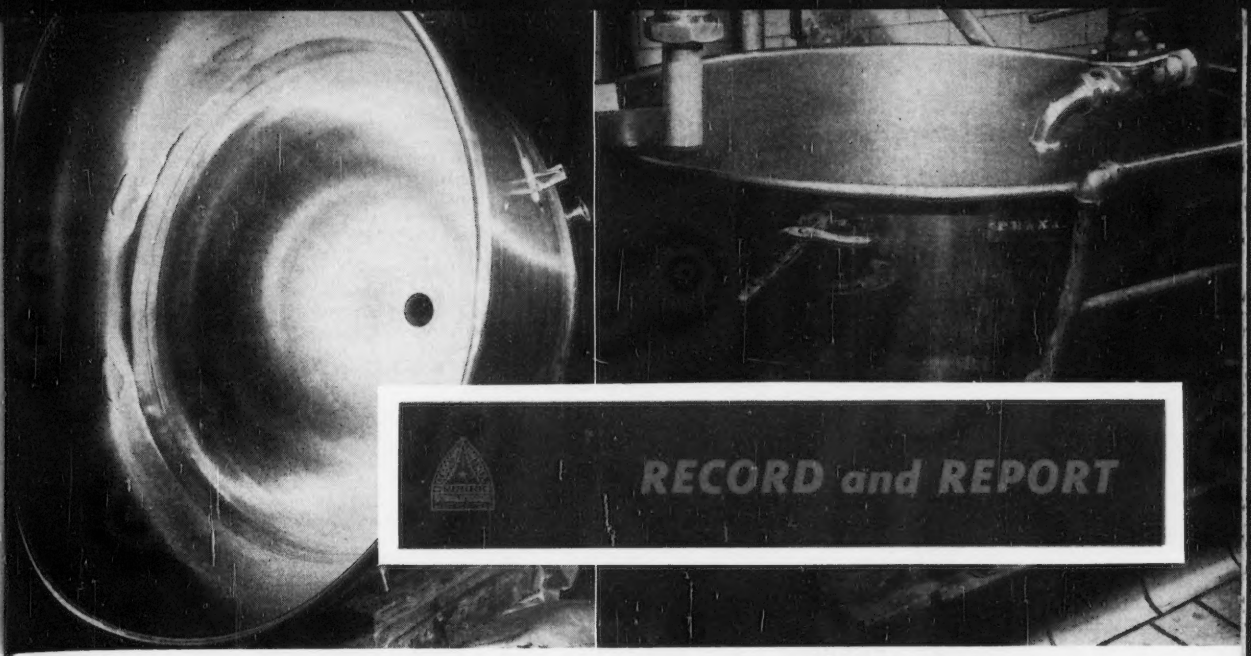
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Tube-Kote Research and Superior Methods Bring You More Durable Coatings and Faster Service... at Lower Cost



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TITANIUM FOOD KETTLE is shown during fabrication at left. The 250-gallon vessel was used in 4000-hour tests at several food processing plants to determine its ability to withstand severe corrosion conditions. Shown at right in service at a food plant, the vessel was reported to be free of corrosion after the tests.

Titanium Used Successfully For Food Processing Vessels

Titanium food processing vessels were tested recently to determine if this material would be feasible for the food industry where certain corrosive solutions pit and perforate stainless steels in a short time.

The 250-gallon titanium vessels were tested under different food preparation conditions by the H. J. Heinz Company in different plants for preparation of pickles, sauces, ketchup and strained foods. In all production runs, the titanium kettles were reported to be unaffected by any of the foods prepared.

Test Program

The test program included those food varieties which have seriously corroded and pitted nickel and stainless steel kettles. Most 300 series stainless steels are corroded by solutions containing sodium chloride and acetic acid—both being common solutions in the food industry, particularly in the processing of pickles. Heinz reports that garlic, especially when fresh, makes the mixture even more corrosive.

Tests included five varieties of food: vinegar for Kosher dill pickels, relish blending, savory sauce, a basic sauce, ketchup and strained and junior foods. Kettles made of Type 316L heat transfer surface corroded badly and pitted through in six weeks in the dill pickle service. In the very corrosive basic sauce service, a nickel heating surface roughens very badly. Ketchup was only moderately corrosive. The other food services were not corrosive but were included to obtain more complete service data on the titanium.

Because acid and salt content and the pH of solutions are important factors contributing to corrosion, chemicals characteristics of the foods in the tests are given in the accompanying table.

Cleaning Solutions

In determining a suitable material for food processing vessels, the effect of various cleaning agents must be considered in addition to chemical attack by food products. During one day's operation, kettles are rinsed after each food batch. Several methods can be used, depending on the product prepared. The simplest method is to scrub the vessel with a nylon brush and hot water. If the food is dried on the vessel or if a stain has developed, certain alkaline cleaners are used to loosen the deposit.

The titanium kettle was given the same cleaning as an adjacent nickel or stainless steel kettle used for the same food product. The kettles were cleaned by the following procedures during the tests.

1. Scrubbed 95 times with a proprietary powder cleaner and a soft brush.
2. Cleaned 160 times by a heavy duty caustic type cleaner, using 4 ounces per 100 gallons of water, heated to 140 F and scrubbed with a nylon brush.
3. Cleaned 30 times with heavy duty caustic type cleaner, using 50 ounces per 100 gallons of water heated to 200 F and held for at least 15 minutes, followed by scrubbing with a nylon brush.
4. Scrubbed 25 times with chlorinated trisodium phosphate, using 50 ounces per 100 gallons of water, heated to 200 F and held for half an hour.

In most of the cleaning procedures, the titanium required less brush scrubbing than the nickel or steel vessels. According to the Heinz report, the titanium vessel was bright and free of any signs of attack after more than 300 cleaning operations with various alkaline cleaners or scouring powders. The titanium did have an evaporation rate slower than similar nickel kettles.

Manufacture of Vessels

Normally, hemispheric shells similar to the food processing vessels are made from seamless, deep-drown section or from welded orange-peel segments. Although there was no information available on deep drawing titanium shapes in the large size required for the kettle, this fabricating technique was chosen.

This decision was made because of the following reasons: (1) past experience in the food industry showed seamless steel design to be more reliable, (2) past experience with preferential corrosion at the welded seams of alloy kettles (although titanium welds had not shown this type of attack), (3) relative simplicity of fabrication and (4) know-how in this type forming would be valuable in other fields where titanium could be used.

Although the inner shell of the kettle is the only surface that contacts the food being prepared, the outer shell or steam jacket also had to be made of titanium so that it could be welded to the inner shell. Ductile welds between titanium and other metals are not obtainable yet.

Inner shell of the kettle was a 45-inch diameter hemisphere drawn from

(Continued on Page 44)

Chemical Characteristics of Various Foods

Food Service	Acidity as Acetic (% by Wt.)	pH	% Salt	Temp., F	Hours in Service
Vinegar for dill pickles	2.0	3.75	5.5—6	140	320
Relishes	2.7—4.2	3.5	1.75—3.5	60–70	830
Savory Sauce	2.6	3.5	3.3	212	65
Basic Sauce	4.4	3.1	6.0	212	1200
Ketchup	1.75	3.85	3.25	212	310
Strained Foods	natural fruit acids	4.0 or lower	none added	....	1400

Roumania Shows Growing Interest in Corrosion Control

Several periodicals published in Roumania have been received by the National Association of Corrosion Engineers in recent months. Roumanian schools, universities and technical organizations have been showing increasing interest in corrosion control literature in recent years. Lack of translation facilities at NACE has prevented an assessment of the contents of the magazines with respect to the degree of interest they are likely to have for corrosion workers. Brief descriptions of some of these magazines follows:

Bulletin of the Polytechnic Institute of Jassy. 480 pages, 6¾ x 9⅜ inches. Vol. IV, No. 3-4, 1958. (New Series.) The contents pages are printed in Roumanian, Russian and French. Content is mainly mathematical, but some articles bear on the physical sciences, others on the biological sciences.

Articles may be in Roumanian, Russian, French or German with a few in English, demonstrating the multilingual capacities of Roumanian scientists. Abstracts of articles usually are presented in at least one language in addition to one in the language of the article, some articles having abstracts in as many as three languages, including English.

"Considerations for the Study of Some Results Obtained in the Study of Vegetable Cancer," by Cristofor Simionescu, corresponding member of the Roumanian Popular Republic Academy, in English, gives results of experiments in this subject.

The volume contains numerous tables of data, and line and halftone illustrations. Complicated mathematical formulae are carefully and precisely printed.

There is a 64-page section devoted to book reviews, mostly of Soviet-bloc origin, but including some of English, American, Australian and other origins.

Chemical Works and Research. 191 Pages, 6½ x 9½ inches. Vol. X, No. 1, 1959. Academy of the Roumanian Popular Republic. Contents pages in Roumanian, Russian and French. Subject matter includes studies with isotopes, calculation of the concentration of hydrogen ions in electrolytic solutions, and others, with emphasis on mathematics. A leading editorial concerns the 15th anniversary of the liberation of Roumania by the Russian army from fascist tyranny.

Applied Mechanics Journal. 169 pages, 6¾ by 9⅜ inches. Vol. 4, No. 2, 1959. No. 3, 162 pages). The foreword states that the magazine publishes original papers in the fields of aeronautics, hydrodynamics, mechanical engineering, general mechanics, civil engineering and measurement technique. Papers are published in Roumanian, French, Russian and English.

Emphasis is on mathematics.

Applied Mechanics. 299 pages, 6¾ x 9¼ inches. Vol. 3, No. 2, 1959. (No. 3 has 233 pages.) Contents in Roumanian, Russian and French. Concerns original papers, short communications and revues of aeromechanics, hydromechanics, mechanical construction, general mechanics, static and dynamic construction, measuring techniques.

Presentation tends to be largely mathematical. There are some line and halftone illustrations.

Two New Plants Being Built

New plants and expansions in production of seamless tubing and vinyl resins and compounds are underway for Seamless Tube Division of Copperweld Steel Company, Shelby, Ohio, and B. F. Goodrich Chemical Company, Cleveland 15, Ohio.

Goodrich Chemical is building a new plant in Watson, Cal., to produce vinyl resins and compounds. Seamless Tube is expanding its facilities in Ohio with new equipment and revamping of present facilities.

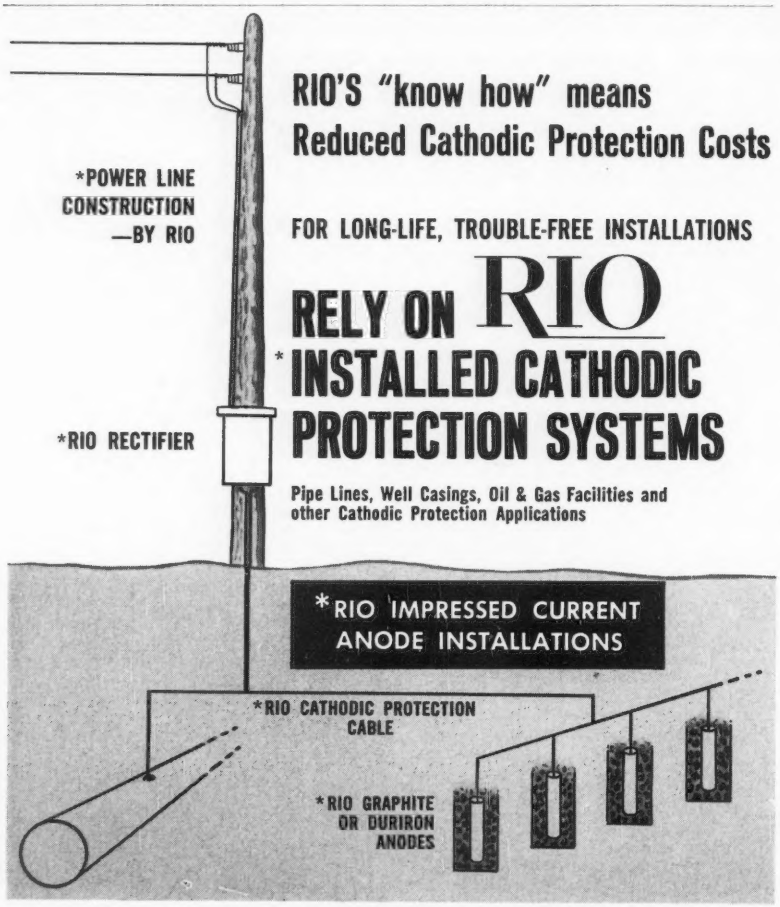
Titanium Used—

(Continued From Page 43)

0.178 thick sheet; the outer shell was a similar shape formed from 0.125-inch sheet. Blanks for both shells were 66 inches square. Republic Steel Corporation supplied the titanium sheet. The B. H. Hubbert Company, Baltimore, Md., developed the deep drawing techniques for the titanium sheet.

Draw ring for the large shell had a 46-⅛-inch ID with a draw radius of one inch. Sheet for the inner shell was Republic Steel's RS-55 grade; the other sheet was RS-70. Blanks were cut with circling shears and carefully edge-around to remove all irregularities.

The 0.178-inch heated blank was drawn to a total depth of 21 inches in four stages. The heated 0.125-inch thick blank was formed to a depth of 23 inches in six stages. Estimated drawing temperatures were 500 to 600 F.



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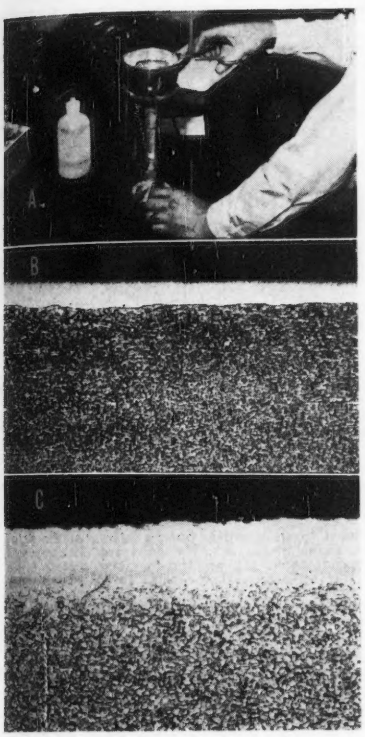
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Platinum Cladding Used for Laboratory Ware



A platinum alloy clad to a corrosion resistant stainless steel has been developed for laboratory ware and also is available in sheet, wire and tubing forms.

The platinum alloy and steel are clad by a patented solid-phase bonding process without diffusion and without use of any brazing alloy or other intermediate material. The clad metal is as corrosion resistant as solid platinum at temperatures to 1000 F and higher, depending on the platinum layer thickness.

Platinum-clad stainless steel laboratory evaporating crucibles and dishes are being produced by the Metals and Controls Division of Texas Instruments, Inc., 34 Forest St. Attleboro, Mass.

About 90 percent of evaporating work is done at temperatures lower than the maximum limit for clad vessels. The 1000 F temperature limit can be raised by increasing the thickness of the platinum layer. All acids used with solid platinum ware can be used in the clad-metal containers.

Standard thickness of the clad-metal ware is 0.035 inches, about four times thicker than solid platinum ware. Life of the clad vessels at temperatures used for evaporating purposes is longer than solid platinum vessels because of the heavier gauge and stronger metal.

To prevent contamination by the outer layer of steel, the clad vessels have rolled edges so that no base metal is exposed to acids near the pouring edge.

PLATINUM-CLAD LABORATORY WARE has been tested to determine its service uses and life. Clad-metal crucibles and dishes were designed to have pouring spouts and rolled top edges to prevent chemical reaction of the stainless steel outer layer with the platinum cladding on the inside. Figure A shows a corrosive chemical being evaporated in a crucible. Figure B is a photomicrograph (500X), showing the absence of diffusion between the platinum alloy and the stainless steel. This was achieved by a solid-phase bonding process used to clad the two metals without use of a brazing alloy or other intermediate material. Figure C, a photomicrograph at 500X shows the diffusion resulting from a test that simulated use for one month at 1550 F. About 15 percent of the platinum layer has alloyed with the steel. This amount of diffusion is allowable and will not affect the crucible's performance.

Technical Topics Set For Early Publication

Development and Characteristics of Reinforced Furane Resin Systems Used in the Chemical Industry, by John Delmonte

Corrosion Control Applications of Electroless Process for Chemical Nickel Plating, by W. J. Crehan

Current Densities to Protect Steel Piling in Fresh Water Tidal Estuaries, by D. B. Bird and H. G. Burbidge

Centralized Chemical Feed to Multiple Cooling Towers, by P. G. Bird and W. B. Harrison

Characteristics of Wood to Handle Chemicals, by Arthur P. Pfeil

Use of Chemical Resistant Masonry, by Robert R. Pierce

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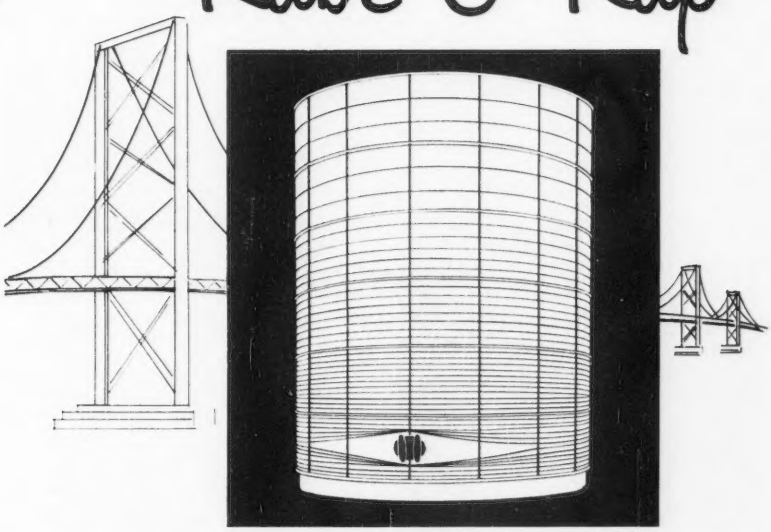
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Metallic Coatings Applied by Vapor and Vacuum

Tungsten Is Plated On Metal Surfaces By Vapor Deposition

High purity tungsten can be plated on metal surfaces by a vapor deposition process developed by the National Bureau of Standards for the U. S. Navy. Gaseous tungsten hexafluoride is reduced with hydrogen by passing it over the heated object to be plated. At temperatures above 300 C, tungsten is deposited on the hot surface. The only other reaction product, hydrogen fluoride, passes out with the excess hydrogen.

Numerous simple and complex surfaces can be coated by this method such as rocket and missile nozzles and jet engine parts. Also, articles can be fabricated of tungsten by this method and may be the only way that complicated shapes of tungsten can be formed.

High Temperature Service

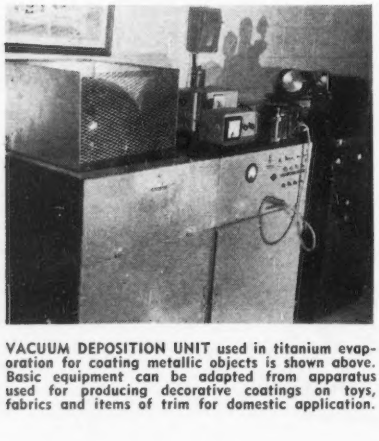
Because of its structural strength at temperatures above 2000 C, high melting point (3140 C), hardness and corrosion resistance, tungsten is a desirable material for high temperature service. Until recently, however, these high temperature properties of tungsten could not be used effectively because the material's brittleness prevented conventional machining and its weight permitted only restricted use in aeronautical equipment. For these reasons, research was begun to develop a practical method for depositing tungsten coatings.

Because tungsten deposition has not been accomplished from either aqueous or organic solutions, deposition from fused electrolytes and the gaseous phase were evaluated. Electrolytic processes were eliminated because the plating rate was too slow, deposition too rough and coatings could not be built up to the desired thickness.

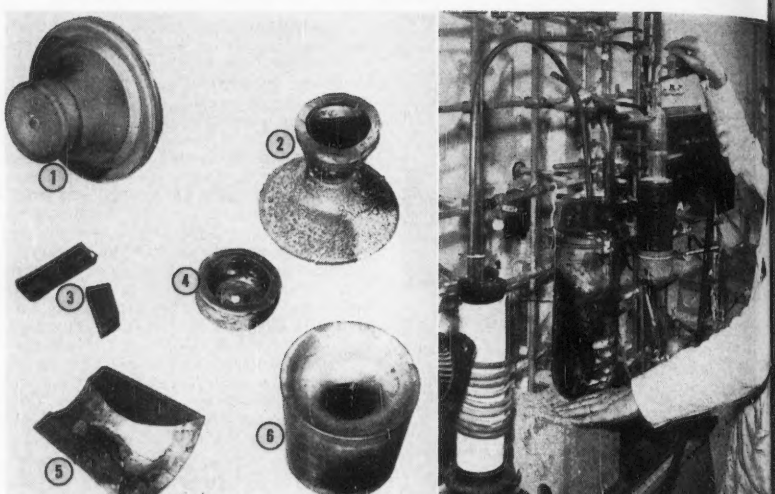
Gaseous Phase Method

Tungsten hexafluoride was selected for use in the vapor deposition process for two reasons: (1) the compound is a gas at room temperatures and therefore convenient to use and (2) tungsten apparently forms only one compound with

(Continued on Page 47)



VACUUM DEPOSITION UNIT used in titanium evaporation for coating metallic objects is shown above. Basic equipment can be adapted from apparatus used for producing decorative coatings on toys, fabrics and items of trim for domestic application.



HIGH PURITY TUNGSTEN PLATING on metal surfaces by a vapor deposition process (shown at the right) can be used to coat simple and complex tungsten forms shown at the left. The parts are (1) component for an electron furnace, (2) thick tungsten rocket nozzle insert, (3) small rectangular pieces, (4) a crucible, (5) cross section of a rocket nozzle and (6) the complete rocket nozzle. To produce tungsten coatings, hydrogen and tungsten hexafluoride are passed over the object to be plated (in ceramic cylinder at left in the laboratory set-up shown above). Hydrogen fluoride, the only impurity formed, is absorbed in the trap (vessel in center).

Titanium Coatings Are Deposited By Vacuum Metallizing Process

A vacuum metallizing process for producing titanium protective coatings has been developed by the Physical Sciences Laboratory in collaboration with the Materials Branch of the U. S. Army Ordnance Tank-Automotive Command at Detroit, Mich. Coatings from 0.5 to 1.5 mils thickness have been obtained.

Although these metallized films are comparable in thickness to commercial electroplated or hot dipped coating standards, special techniques were applied to the coating process to obtain an adherent, uniform, compatible coating, especially on ferrous base metals. In laboratory practice, thick titanium films are formed by evaporation from 0.05-inch thick tungsten boats heated electrically.

A preliminary vacuum degassing of the base metal provides clean, gas-free surfaces for the titanium vapor. No other pre-cleaning treatment or surface conditioning is necessary because coating adhesion is not dependent on a mechanical surface bond. After the coating is accomplished, the composite titanium film and base metal are vacuum diffused to eliminate porosity and to increase coating adhesion. This technique gives a non-porous film with excellent coverage and tenacity.

Extremely resistant to corrosion, titanium coatings on ferrous metals have passed the 200-hour salt fog exposure test prescribed for corrosion resistant materials in Federal Specification QQ-M-151a.

The basic equipment needed for commercial production of vacuum metallized titanium is adaptable in the form of apparatus used for producing decorative coatings on toys, fabrics and items of trim for domestic application.

Army Ordnance was particularly interested in the titanium coatings to reduce galvanic corrosion between anodic titanium and iron or other structural metals used in vehicle parts. Undesirable galvanic couples occur when small iron brackets or fixtures are attached to large titanium structural components. In these applications, titanium coatings on the iron brackets will eliminate the hazards of galvanic corrosion.

Many applications in industry may be found where thin coatings of corrosion resistant metal are needed. Many parts coated with electroplated nickel could be protected more effectively by metallized titanium films deposited by this vacuum method. Considerable savings might be made by substituting titanium coated mild steel for solid or clad titanium now necessary because of the lack of a satisfactory method of depositing adherent thin films.

NBS Has 600 Standard Material Samples Available

The National Bureau of Standards is now distributing 60,000 samples of standard materials a year to other laboratories for use in controlling chemical processes and in maintaining accuracy of apparatus and equipment. A total of 600 different standard materials are available, including chemicals, ceramics, metals, ores and radioactive nuclides. A complete list is available from the Standard Samples Clerk, NBS, Washington 25, D. C.

Tungsten Is –

(Continued From Page 46)

flourine—WF_6. Thus the reactions are uncomplicated by side reactions.

The equipment required is simple. The object to be plated is placed inside a cylinder of ceramic material and the air pumped out and replaced by hydrogen. A controlled temperature induction furnace heats the object to about 650 C. Then the two gases, tungsten hexafluoride and hydrogen, are passed through ordinary glass flowmeters into the reaction chamber. When the gaseous mixture comes in contact with the heated object, tungsten is deposited on the hot surface. By-product of this reaction, hydrogen fluoride, is absorbed in a suitable trap.

Nature of the Deposit

Spectrochemical examination shows the tungsten obtained by vapor deposition is purer than ordinary commercial tungsten. Silicon is the only impurity present in a concentration of 0.1 percent or more. The deposited metal has the theoretical density of pure tungsten which is higher than that of tungsten compacts produced by powder metallurgy or by flame spraying. That is, plating eliminates the tiny holes between tungsten particles resulting from the other two processes. Hardness of the deposits obtained at 650 C is about the same as ordinary commercial tungsten (475 Vickers Hardness Number).

Adhesive band between the tungsten coating and various basic materials was investigated. Adhesion to molybdenum and to nickel was good, somewhat weaker to copper and rather poor to iron. The tungsten-graphite bond was about as strong as the graphite itself. Tungsten deposited on ceramics also showed good bonding.

Deposits 1/16-inch thick were obtained by vapor deposition in an hour. In this same time only 0.001-inch thickness of tungsten could be plated from fused salts melts in electrodeposition. Maximum thickness obtainable by the vapor method has not been determined but would depend on the permissible roughness of the surface. Because of its rapid deposition, this process could be useful for continuous production of tungsten coated strips or wires.

Uniform Distribution of Coating

Uniform distribution of tungsten over an irregularly shaped object is a problem that must be solved for each item to be coated. Deposition is more uniform over the exterior of an object if plating is conducted in a chamber much larger than the object. Because tungsten deposition occurs in greatest quantity where the gases first strike the hot object, better distribution probably can be obtained by passing hydrogen through the main opening in the chamber and leading the WF_6 to various parts of the object by a system of injector tubes.

To obtain a desired tungsten shape without the base metal underneath, the metal form is chemically dissolved after deposition. The method of separating the tungsten depends upon the other metal used.

•

There were 311,758 stockholders in one U. S. steel company in 1958. These stockholders included men and women from all walks of life in every state in the nation.

Oil Well Paraffin Deposits Prevented by New Method

New method to prevent paraffin deposits in oil wells has been developed in which all metal parts subject to treatment must be cleaned of any oil, wax and paint. After this cleaning step, a special combination of chemicals is applied to make the metal parts water-wet and thus prevent the adhesion of paraffin particles to the clean, water-wet surfaces. Chemical treatment is done the same way as other down-hole treatments such as demulsifying agents and corrosion inhibitors.

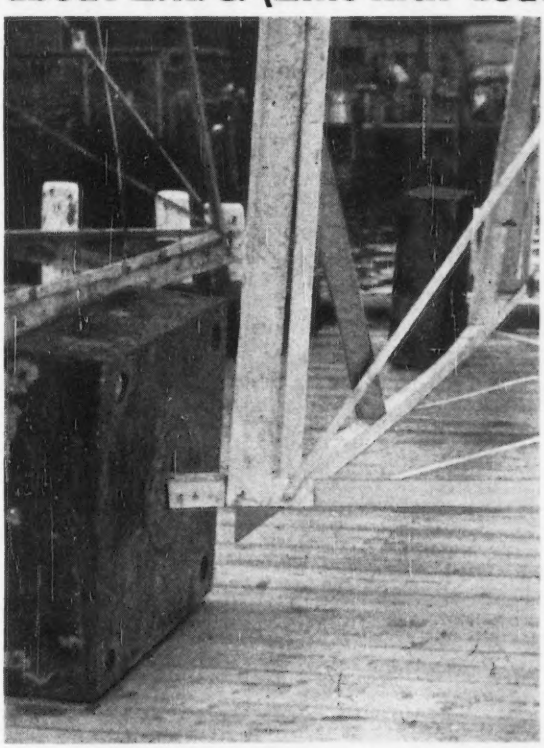
Chemical formulation is easily handled, nonflammable and presents no toxicity problems, according to the developer, Dowell, Tulsa, Okla. Also it is harmless to refinery processes.

Geodesic Dome to Be Gold Anodized Aluminum

Gold anodized aluminum tubing will be used for a 90-foot diameter geodesic space frame in Tampa, Fla. The golden dome structure will serve as a sanctuary for large birds in Anheuser-Busch's gardens in the Tampa Industrial Park.

Graver Tank & Mfg. Co., East Chicago, Ind., will construct the Garverdome of the anodixed aluminum tubing, 3 inches in diameter and 1/8-inch thick. The structure will be covered with a 2-inch square mesh, also of gold anodized aluminum.

No glamour—just facts about Z.R.C.® (Zinc Rich Coating)



Angle iron in this picture was treated with Z.R.C. Heavy steel base was not—here's the result after several months at sea.

This is what the OCEANOGRAPHIC INSTITUTE, Wood's Hole, Mass. has to say . . .

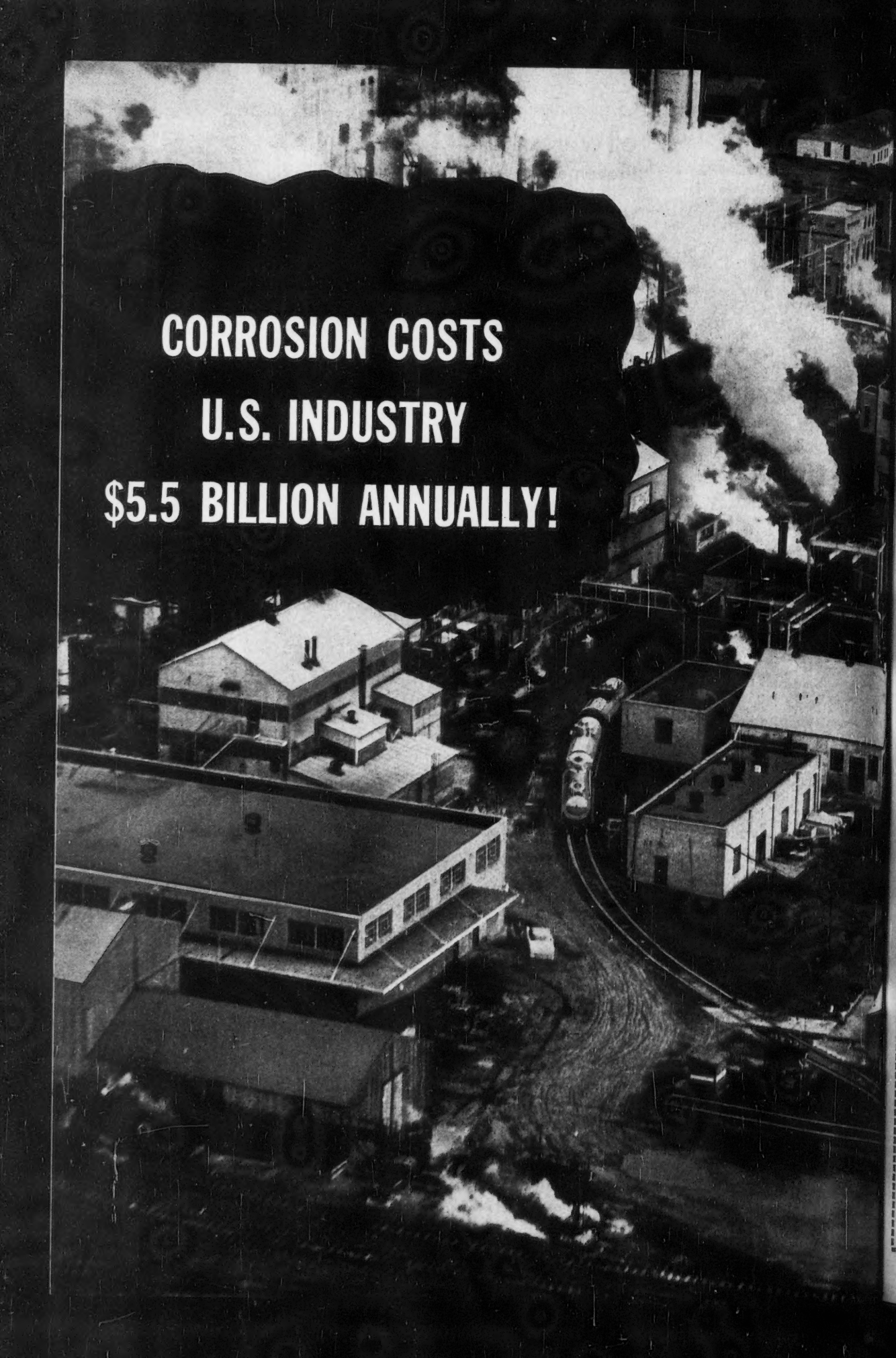
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For additional facts and technical information . . .

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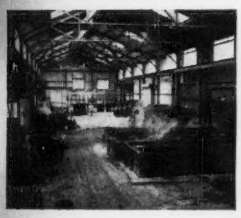
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PITT CHEM TARSET®



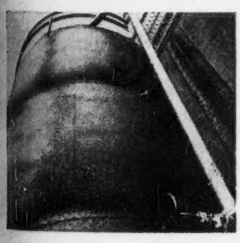
The original, patented coal tar-epoxy coating, Tarset is being used throughout industry to solve corrosion problems that were once considered "impossible." This revolutionary cold-applied coating is ideal for the protection of underground storage tanks, pickling tanks, high temperature steam lines, waste tanks and similar equipment.

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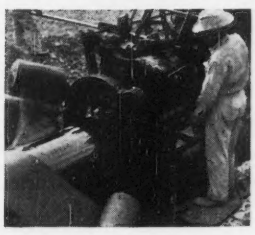
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CATHODIC PROTECTION

By
LINDSAY M. APPLEGATE
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244 pp., 67 illus., $9.00

Here is a complete and practical guide to combatting corrosion of underground and submerged structures by the electrochemical means of cathodic protection.

The book explains the chemistry of the corrosion of metals when surrounded by an electrolyte such as water or moist earth and details the practical methods of applying electric currents to mitigate damage under these conditions.

Here is all the information you need to select and use the necessary instruments, make field surveys and tests, interpret results, design adequate systems, and carry out the construction of cathodic protection installations. Practical "how-to-do-it" examples are shown that can be followed in the protection of submerged pipelines, cables, aviation fuel tank and line systems, lock and dam gates, ships in storage, and other underground and submerged structures.

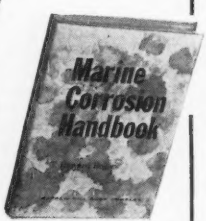
Emphasized in particular are the applications of active metals for sacrificial protection, and the use of inert metals connected to external circuits for inert anode protection of iron and steel.

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MARINE CORROSION HANDBOOK

By
R. HOWARD ROGERS
Naval Research Establishment, Halifax, N. S.

320 pp., 48 illus., $12.50



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BOOK NEWS

Structure and Properties of Thin Films. Editors C. A. Neugebauer, J. B. Newkirk and D. A. Vermilyea. 561 pages, 8 x 11 inches, cloth. December 18, 1959. John Wiley & Sons, Inc., 440 Fourth Ave., New York 16, N. Y. Per copy, $15.00.

Proceedings of an international conference held at Bolton Landing, N. Y., September 9-11, 1959, sponsored by the Air Force Office of Scientific Research, Air Research and Development Command and General Electric Research Laboratory, Schenectady.

The 41 papers are arranged under the following topical headings: Formation of thin films, mechanical properties, electrical properties, magnetic properties, chemical interactions at surfaces, theory of surfaces, a panel discussion. Discussions about each paper are included as recorded.

Examples of the research underway by participants in the conference are given in the papers. These include studies of phase transformations, chemical reactivity, superconductivity, electrical resistance, Hall effect, magnetic properties and magnetoresistance. Also properties of films not exhibited in bulk form. Other papers are concerned with direct observation of defects, structure studies, measurements of specific surface area and observations of the state of stress. Several papers deal with surface chemistry.

A few papers on theory cover epitaxial relationships, the manner in which stresses are introduced and interactions of atoms and surfaces.

Photolithographed in large size typescript, the book is abundantly illustrated and has a subject and author index.

Corrosion Prevention. 431 pages, 8 x 10¼ inches, loose-leaf, paper covers. 7 December 1956, PB 151756. Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. Per copy, $6.

This is Part M of Maintenance and Operation of Public Works and Public Utilities prepared by Office of the Chief of Civil Engineers, Bureau of Yards and Docks, U. S. Navy. Printed from typescript.

An extensive, thoroughly illustrated exposition of the corrosion problems associated with the structures covered and necessary remedial measures. The book assumes limited knowledge of the corrosion reaction and gives a brief, but thorough exposition of the nature of the corrosion reaction, and the principal reasons corrosion occurs. An extensive list of definitions of corrosion terms is helpfully included.

Topics covered include dissimilar metal connections, differential environments, chemical attack, stray currents, microbiological corrosion, atmospheric corrosion, available methods of control, use of non-metallic materials, coatings, claddings and coverings, passivity, changes of environment, material selection, cathodic protection, economics, field surveys, marine corrosion, electrical grounding, electromagnetic interference, stress and miscellaneous types.

One of the best features of the book is the extremely detailed and comprehensive table of contents. This makes the book especially useful for the practicing corrosion engineer concerned with the structures and environments considered. Sufficient information is given on the fundamentals of the reactions involved to facilitate understanding of the reasons for the remedial measures.

The section on economics, while designed with military establishments in mind, includes examples of power costs, formulae for computing annual costs, initial cost and salvage value, straight line method of depreciation and other useful concepts.

Corrosion of Chemical Apparatus. By G. L. Svartz and M. M. Kristal. 256 pages, 6 x 9 inches, cloth. (Translated from the Russian.) 1959. Consultants Bureau Inc., 227 West 17th St., New York 11, N.Y. Chapman & Hall, Ltd., London. Per copy $7.50.

This monograph on the corrosion and stress corrosion of metals and alloys used in chemical plant construction was originally published in Russian in 1958 by the State Scientific Technical Press for Machine Construction Literature, Moscow. It is designed for all persons concerned with chemical plant corrosion problems.

The authors first consider the theoretical factors involved in corrosion cracking and then consider the corrosion cracking of carbon and low alloy steels. The section on stainless steels covers chromium and chromium nickel steels. The portions dealing with chromium-nickel austenitic steels under static stress include such topics as effect on structure of steel, effect of chemical composition of steel, heat treatment, stress, effect of the nature of the medium with respect to its contents and their concentrations.

The section on non-ferrous metals and alloys covers aluminum, copper and copper alloys, nickel and nickel alloys, magnesium, lead, titanium, and others.

The section on preventive measures includes material selection, construction methods, techniques, stress relief, electrochemical measures, with special attention to protection against intercrystalline corrosion. The final section is on testing methods.

Of the 219 references only a small percentage are to material published outside of the Soviet orbit. The book is thoroughly illustrated and contains many tables and graphed data.

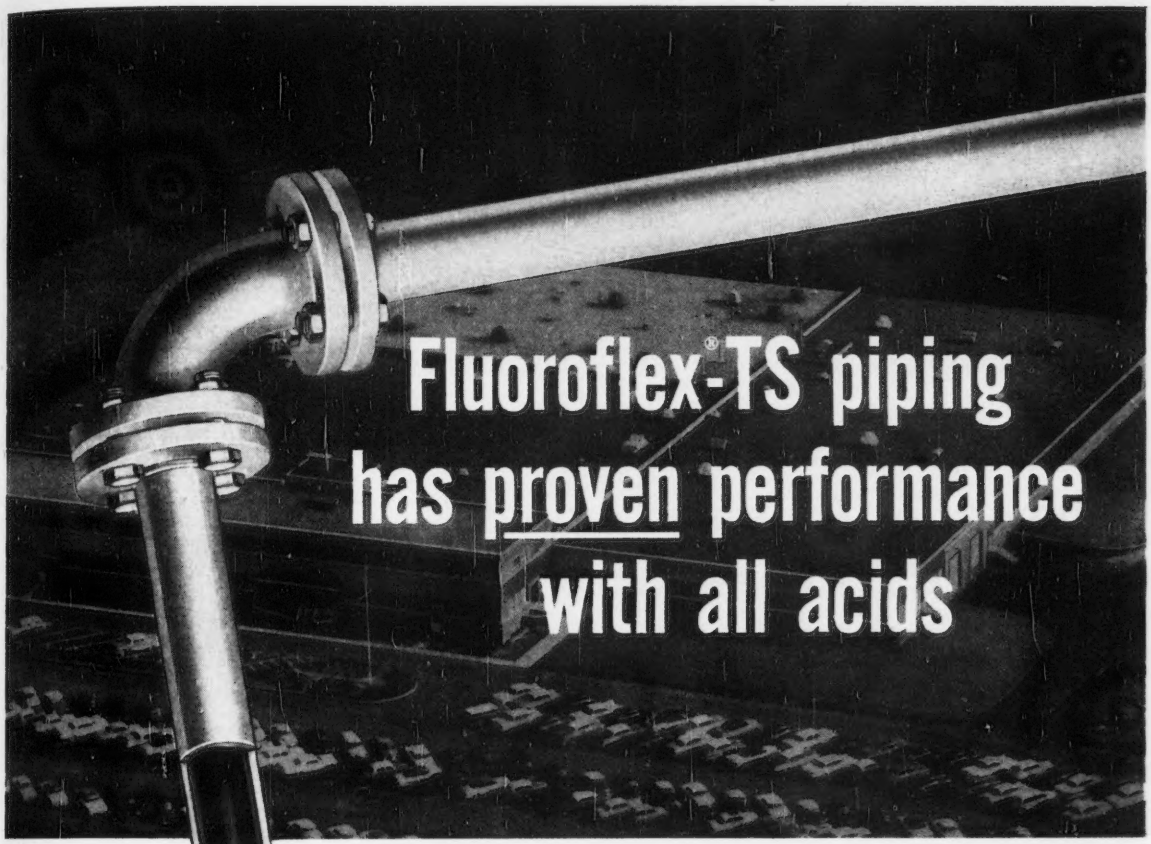
A Digest of State Air Pollution Laws. By Samuel M. Rogers and Sidney Edelman. 117 pages, 8 x 10¼ inches, paper. 1959. Public Health Service Publication No. 711. Superintendent of Documents, U. S. Government Printing Office. Per copy 75¢

The laws concerning air pollution for all of the United States and its possessions are presented in digested form.

Welding Ferrous Materials for Nuclear Power Piping. 14 pages, 6 x 9 inches, paper. 1959. American Welding Society, 33 W. 39th St., New York 18, N. Y. Per copy, $1.50.

A report by AWS committee on piping and tubing. Contents include general considerations, materials, welding procedures, root pass welding, fabrication quality control and acceptance inspection; welder performance and procedure qualifications.

Clear illustrations are included of the operations discussed.



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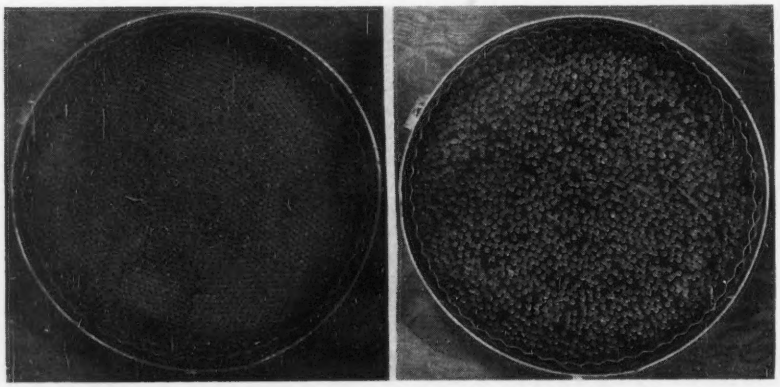
INCREASED MANUFACTURING FACILITIES now in operation reflect the growing demand for Fluoroflex-TS piping. Corrosion engineers and process designers can depend on Fluoroflex-TS piping backed with full production operations by the company that originated Teflon hose and pioneered the development of Teflon lined pipe.

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BEHAVIOR OF ATOMS is being studied with a newly developed model which simulates atomic structure by means of glass beads on a flat jiggling plate. Kept in constant random motion by a motor beneath the plate, the beads at right above illustrate atomic behavior in a liquid—loosely packed with no visible pattern arrangement. At left, a solid has formed with the grain boundaries beween adjacent groups of atoms clearly visible.

Behavior of Atoms in Liquids Simulated by Glass Bead Model

How atoms move in gases, liquids and solids is being demonstrated in a mechanical model recently developed. The new device, in which several hundred glass beads jostle each other on a flat vibrating platform, is being used to help researchers solve one of modern science's more important problems: how atoms move in liquids.

Glass beads are used in the model as stand-ins for atoms. The beads are poured onto a round glass plate with a fence on the outside edge to keep them from rolling off. The plate, suspended from springs, is vibrated constantly by a motor mounted beneath, making the beads behave as atoms are believed to do. This behavior duplicated in the model can be photographed and studied.

Scientists are particularly interested in atom behavior because properties of matter result from the interaction of many atoms. If these interactions can be calculated, the behavior of matter can be predicted. However, precise calculations are extremely difficult. The model may suggest how these calculations can be simplified.

The model duplicates the ceaseless random motion that atoms are believed to be in at all times. Many atomic interactions believed to occur in actual matter can be illustrated by the model.

When only a few beads are poured onto the vibrating plate, they behave as atoms do in a gas—moving rapidly over relatively long distances between collisions with each other and arranging themselves in no particular pattern. As more beads are added to the vibrating plate, they travel shorter and shorter distances although they are still moving rapidly and still showing no pattern. This condition represents atomic behavior in a liquid. As the number of beads is increased, they eventually freeze into a solid, becoming confined most of the time to a particular position. A distinct pattern of several large areas is separated by lines well known to corrosion engineers as grain boundaries.

Motion pictures taken of the model in action permit the study of many aspects of tomic behavior including the question of how the large areas of close-packed atoms enlarge themselves, pushing their grain boundaries out until neighboring small areas disappear. One drawback of the model is that the glass beads do not attract one another as real atoms are supposed to do. Inside a solid material, however, such atomic attractions largely cancel each other out.

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PERIODICALS

U. S. Government Research Reports. Monthly. Published by Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C. Domestic subscriptions, yearly, $6. Foreign subscriptions, $9. Single copies 60 cents. In U. S. make checks payable to Superintendent of Documents; Outside U. S. remittances payable to OTS, U. S. Dept. of Commerce.

A magazine abstracting reports issued by government research agencies or by agencies doing research under government contract. A wide variety of information is covered, from Astronomy to Special sciences. Of particular interest to persons active in the corrosion-control field will be the sections devoted to chemistry, engineering, machinery, materials, mechanics, metallurgy, physics, the nuclear sciences.

A source, subject and number index are included.

Russian Articles and Books Translated by U. S. Agencies

Eighteen Russian technical articles and books translated by U. S. government agencies have been published for sale to the public. One of these is an index of scientific and technical periodical literature in Russia, said to be the most complete Soviet refrence publication on periodicals.

The translations can be ordered from the Office of Technical Services, Business and Defense Services Administration, U. S. Department of Commerce, Washington 25, D. C. Also available are three translated reports on Russia's Committee on Determination of Atmospheric Pollutants.

New Research Center

Metallurgical, corrosion and chemical laboratories will be included in the 1½ million dollar research center to be built by the American Brass Company at Waterbury, Conn. Construction will begin this summer, and completion is scheduled for 1961.

Seminar in India

A seminar in electrochemistry has been scheduled for March 21-25 by the Council of Scientific and Industrial Research, New Delhi, India. It will be held at the Central Electrochemical Research Institute at Karaikudi, India.

NEW MATERIAL
for your tough
insulating and
refractory jobs



FOAMSIL®

the acid-proof insulating refractory

The photograph above illustrates one critical application in which material failure would be, to say the least, unfortunate. Undoubtedly *you* have many severe plant or process operations where insulation or refractory failure would be equally unfortunate. It is for just such applications that Pittsburgh Corning has developed its new acid-proof insulating refractory—called FOAMSIL.

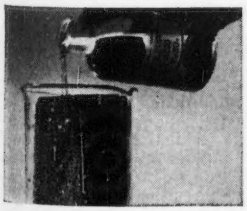
FOAMSIL is 99% pure silica expanded to form millions of airtight cells. These cells create excellent insulating value, and since the cells are sealed against moisture, that insulating value remains constant.

Because FOAMSIL is composed of silicon dioxide, it cannot oxidize further—thus withstands high temperatures. It won't react with common acids and has a high melting point. It is incombustible, will not lower the flash point of hydrocarbons, will not thermal shock and is dimensionally stable. It is easy to cut and fit.

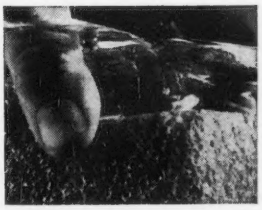
Among potential applications for which FOAMSIL should prove ideal are these: acid-proof tank linings . . . precipitators . . . furnace linings . . . lead acid chambers . . . concentrator towers . . . stack and flue linings . . . reradiating shields . . . environmental test chambers . . . insulation for underground piping. Your own insulating and refractory requirements will undoubtedly suggest a host of other uses for FOAMSIL.

Write today for a free sample of FOAMSIL and complete descriptive literature. Find out how to put this unique new insulating refractory to work to improve *your* operations. Write Pittsburgh Corning Corp., Dept. CO-30, One Gateway Center, Pittsburgh 22, Pa.

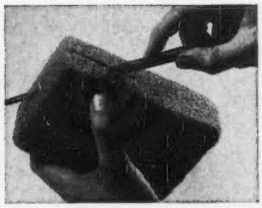
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SIZES AND SHAPES

FOAMSIL is ideal for temperature ranges from −450°F to +2200°F continuous. It is available in flat blocks up to 3 inches thick measuring 11″ x 17″ or 17″ x 22″ . . . and in various standard or special shapes including internal or external pipe insulation, curved sidewall segments, and fitting covers.

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Equipment Services NEW PRODUCTS Materials Literature

Adhesives

Acton Fluoroetch Solution, an activated form of sodium in solution, is designed to make fluorocarbon polymers bondable to other materials. It reacts with fluorocarbon resin extracting fluorine atoms at the surface with the formation of a carbaceous film which is compatible with most adhesives. It is available from Acton Laboratories, 1180 Raymond Blvd., Newark 2, N.J.

•

Polyvinyl Ethyl Ether Resin production has been increased at the new South Charleston, W. Va., plant recently enlarged by Union Carbide Plastics Company, Division of Union Carbide Corporation. This resin is the binding force in the adhesives that maintain permanent tackiness and bonding strength.

Coatings, Organic

Two New Truscon Coating Systems are a chlorinated rubber base coating and a Devran epoxy primer and finish coat. The rubber coating is designed for exposure to sulphuric acid of 75 percent or higher concentration and to concentrated nitric and phosphoric acid. The epoxy system is designed for exposure to active solvents such as ketones, esters and alcohol ethers. The two systems were developed by Truscon Laboratories, Industrial Maintenance Division of Devoe & Raynolds Co., Inc., Detroit 11, Mich.

•

Pre-Coated Metal is being used for portable television cabinets. A special vinyl coating which simulates morocco-leather finish is applied before the metal is formed. Difficult 90-degree bends and 180-degree seams have been made without damage to the coating. Back side of the cabinets receive a clear coating to prevent corrosion.

Instruments

Industrial pH Amplifier with full temperature compensation is described in Data Sheet EIL 28 AM published by Milton Roy Company, 1300 E. Mermaid Lane, Philadelphia 18, Pa. This pH meter is specifically engineered for measurement of pH in industrial manufacturing processes. The temperature compensation makes it possible to set up the instrument in buffer solutions at one temperature and to operate at any other temperature without requiring further adjustment or correction to readings. This non-indicating unit provides an electrical output accurately proportional to pH which is suitable for operating most potentiometric or current operated recorders and controllers.

•

Electrospec 200, a wide spectral range electron spin resonance spectrograph, is being produced by Elion Instruments, Inc., 430 Buckley St., Bristol, Pa. Designed as a basic laboratory research and industrial analytical instrument, the Electrospec 200 is claimed to be suitable for studies involving metals, water based materials, semi-conductors and non-conductors.

•

Du Mont Model 425, a high frequency general purpose oscilloscope, features a direct digital reading system. Manufactured by Allen B. Du Mont Laboratories, Inc., Clifton, N.J., the instrument can be used in manufacturing and process control.

•

Magnox Analyzer, an instrument for continuous measurement of oxygen in gases manufactured by Milton Roy Co., 1300 E. Mermaid Lane, Philadelphia 18, Pa., is designed with automatic temperature compensation without the use of chemicals and moving parts. It provides data for efficient fuel burning, economical ceramic firing and close control of kiln atmospheres. It also measures the purity of oxygen and nitrogen.

Metals, Exotic

Erbium Metal Data Sheet has been published by Nuclear Corporation of America's Research Chemicals Division, 170 West Providencia St., Burbank, Cal. Data refers to properties of this rare earth metal including melting and boiling points, crystal structure, atomic radius and magnetic movement.

•

High Purity Vanadium Metal is being produced by Oregon Metallurgical Corporation, Albany, Oregon. Vanadium is characterized by a high melting point, excellent resistance to corrosion by salt water and most chemicals and the ability to pass moving neutrons without impeding them. Oregon Metallurgical's production of vanadium is claimed to be at least 99.8 percent pure and able to be wrought or worked into sheet or other forms.

Metals, Ferrous

Extruded Stainless Steel Shapes are being produced by H. M. Harper Company, 8200 Lehigh Ave., Morton Grove, Ill., by use of a new furnace to melt the steel. The shapes are extruded by the Ugine-Sejournet patented process which employs molten glass as a lubricant. A brochure describing the company's extrusion activities is available.

•

Cast Steel with lower sulfur content is possible through a new technique developed by General Electric Research Laboratory, Schnectady, N.Y. Sulfur reduction increases the impact strength of the cast metal at low temperatures and reduces hot tearing during casting, according to the GE laboratory. Sulfur content of 0.012 percent is considered good in conventional steel casting, and this has been reduced to 0.008 percent with induction stirring equipment. GE claims its new technique has reduced sulfur content to as low as 0.003 percent.

Metals, Non-Ferrous

Nickel Supply has been expanded with the opening of a new mining project at Thompson, Manitoba, Canada, by International Nickel Co., Inc. By 1961 the mine will be producing 75 million pounds of nickel a year. The producting shaft has been sunk to a depth of 2100 feet.

•

Molybdenum and molybdenum alloys' resistance to corrosion is described and tabulated in a publication "Corrosion Resistance of Molybdenum and Molybdenum-Base Alloys," by R. R. Freeman and J. Z. Briggs. The publication is available on request from Climax Molybdenum Co., 500 Fifth Ave., New York 36, N.Y.

Pipe

Hi-Flo Steel Water Pipe, manufactured by Smith-Scott Co., Inc., P. O. Box 3033, Riverside, Cal., is described in a booklet available from the company. Included are type of pipe manufactured, friction charts, working pressure charts and photographs showing manufacturing and coating operations.

•

Stainless Steel Tubing booklet available from Allegheny Ludlum Steel Corp., Oliver Bldg., Pittsburgh 22, Pa., gives details on various sizes, grades, design data, corrosion resistance and other information on welded and seamless stainless steel tubing. More than 25 tables are included.

Plastics

Six-Inch Vinyl Pipeline is being used as an ocean water intake at the Massachusetts State Research and Lobster Station on Martha's Vineyard Island. Extending 250 feet from the station to the sea, the pipe brings salt water to the station for use in fish propagation. The pipe was made of Geon vinyl, a product of B. F. Goodrich Chemical Company, to eliminate salt water corrosion. Periodic cleaning is required to keep the pipe free of sea mussels and other foreign matter.

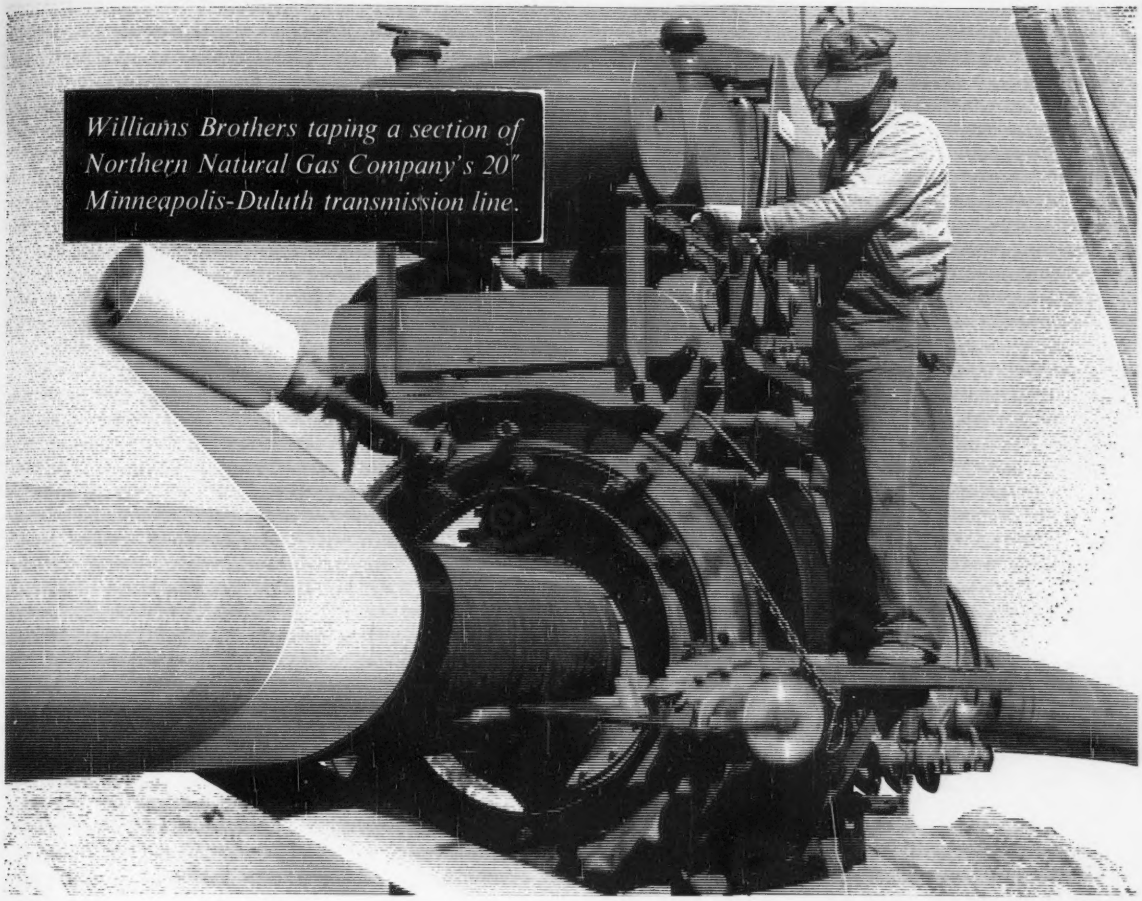
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Teflon 100, a new fluorinated ethylene propylene produced by Du Pont, can be extruded or molded in thermoplastic processing equipment. It was developed to supplement Du Pont's tetrafluoroethylene resins which must be processed in a manner similar to powdered metals. The new resins are claimed to be immune to chemical attack, have excellent electrical insulating, anti-stick and frictional characteristics and will not absorb moisture.

•

Paper Coating designated DGDD-7401 Natural is a high density polyethylene extrusion coating compound developed by Union Carbide Plastics Company, division of Union Carbide Corp., 420 Lexington Ave., New York 17, N.Y.

(Continued on Page 56)



POLYETHYLENE TAPE by SEAMLESS

used for coating 53 miles of 20" main line in Northern Minnesota

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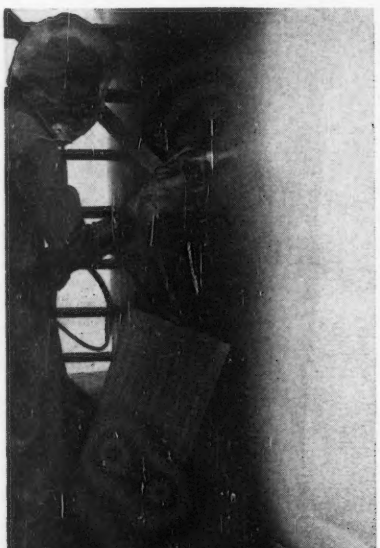
- Actual construction time, 31 days.
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NEW PRODUCTS

(Continued From Page 54)

The material can be extruded onto paper at good commercial speeds in coating weights as low as six pounds per ream. Formerly, 20 pounds per ream was the lowest practical coating weight possible with high density polyethylene.

•

Fluorel KF-2141 Elastomer now has improved workability and is rated for service above 400 F, according to the manufacturer, Minnesota Mining and Manufacturing Co., 900 Bush Ave., St. Paul 6, Minn. Fluorel elastomer is used for O-rings, gaskets and seals for high temperature systems conveying acids and bases, solvents and oils and fuels. It is being used in the aircraft, missile, automotive and chemical industries.

•

Ren Shape, an epoxy resin based paste material produced by Ren Plastics, Inc., 5422 S. Cedar Road, Lansing 9, Mich., is designed for use in the home and industry as a repair material, pattern mold and as a filler and leak repair. The two-part resin-hardener is easily mixed and can be applied with brush, spatula or other handy tools, according to the manufacturer.

•

Hetron Polyester Resin, a chlorinated polyester developed by Durez Plastics Division of Hooker Chemical Corp., North Tonawanda, N.Y., can be fabricated into such laminated products as storage tanks, piping, scrubbers, columns, ductwork and similar processing units or plant equipment. It offers good advantages in temperature and chemical resistance, according to the manufacturer.

•

Nylon Pressure Tubing in a variety of diameters and wall thicknesses is available from United States Gasket Company, Plastics Division of Garlock Packing Co., 440 Main St., Palmyra, N.Y. The tubing is designed for use in high pressure fluid and pneumatic service and in applications where friction is a problem and lubrication impossible or impractical.

•

Nowery J. Smith Company, P. O. Box 7398, Houston, Texas, has acquired exclusive right to apply Goodyear's Plioweld in the Gulf Coast area. Plioweld is a special rubber lining and covering designed to protect metals from corrosion and abrasion.

•

Rigidon, a glass reinforced plastic for ducts, fittings, hoods, stacks, covers and tanks is described in Bulletin B-500 available from Heil Process Equipment Corp., 12901 Elmwood Ave., Cleveland 11, Ohio. Included are standard sizes, specifications and chemical resistance data. Also includes information on Heil's Rigivin, a rigid vinyl material.

•

Corocrete, a specially formulated corrosion resistant material for concrete floors, is produced by Ceilcote Company, 4832 Ridge Road, Cleveland 9, Ohio. Available in a variety of colors, the material is designed to replace paints having low abrasion resistance where application requires resistance to corrosive fluids. It is composed of thermosetting resin, a hardening agent and a special aggregate.

Process Equipment

Serfass Hydrogen Purifier, using a patented palladium-silver alloy that is permeable only to hydrogen and deutrium, is designed to separate hydrogen from other gases under pressures to 150 psi. The alloy does not form a structurally weak Beta phase as does pure palladium when cooled in hydrogen. No special precautions are necessary to prevent embrittlement, and there is no need to purge the purifier with an inert gas or to evacuate it, according to the manufacturer, Serfass Corporation, 1300 East Mermaid Lane, Philadelphia 18, Pa.

•

Storage Tank Operation Safety is included in Technical Bulletin CP-1001 available from Vapor Recovery Systems Co., 2820 North Alameda St., Compton, Cal. Designed to give a better understanding of factors which influence product evaporation rates, the bulletin includes a section of selection of corrosion resistant materials.

•

Chemstor Glasteel Tanks with glass inside and steel outside are described in a new bulletin available from Pfaudler Co., division of Pfaudler Permutit, Inc., 1097 West Avenue, Rochester, N. Y. The tanks are designed for storing corrosive or sticky products that must not be contaminated. Bulletin includes descriptive data, typical applications and physical specifications of the tanks.

Pumps

Oliver Diaphragm Slurry Pump has been designed to meter slurries with 60 percent solids at a controlled volume accuracy of plus or minus 2 percent. Compressed air operation eliminates motor, stuffing box and leaky packing, according to the fabricator, Milton Roy Company, Philadelphia, Pa.

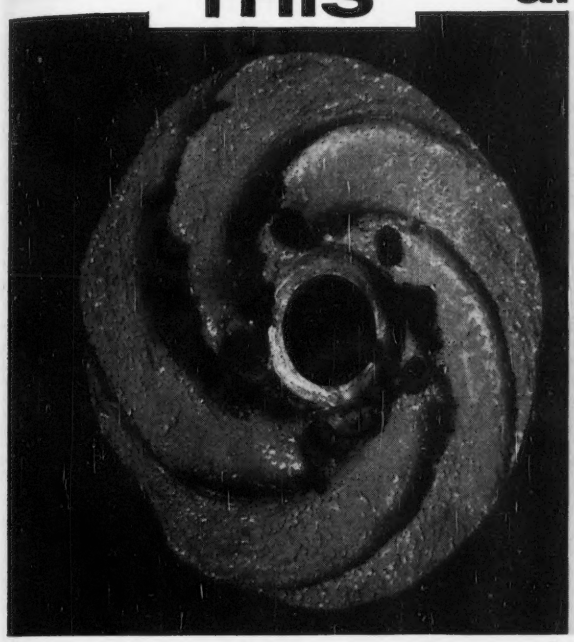
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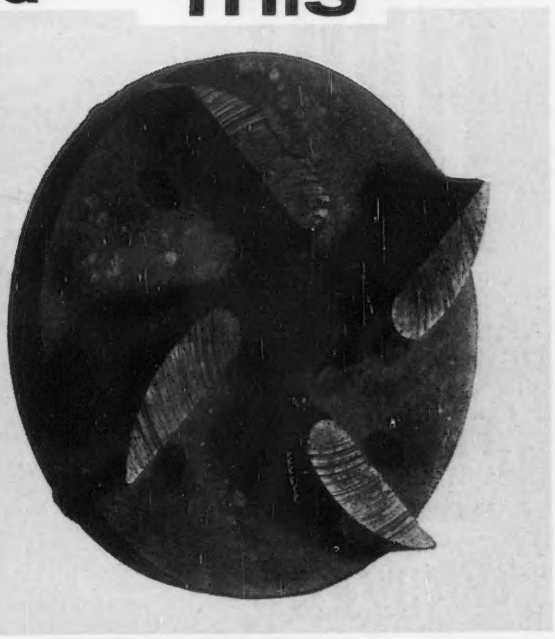
Dynapump Model 520D, a compact pump claimed to be leak-proof, has been developed by the Dynapump Division of Fostoria Corp., P. O. Box 35-3, Huntingdon Valley, Pa. Constructed entirely of corrosion resistant material, it offers the advantages of canned pumps, weighs less than seven pounds and can pump over 800 gallons per hour. Because rotor, shaft and impeller form a single assembly enclosed or "canned" in a stainless steel cylinder, stuffing box and mechanical seals are eliminated. The pumped fluid circulates in the rotor chamber and acts as bearing lubricant and motor coolant.

Rectifiers

Eicor Incorporated, 1501 W. Congress St., Chicago 7, Ill., has released a brochure titled "The Eicor Story," which gives background information on the company and its production plant at 517 W. Walnut St., Oglesby, Ill. The company has begun manufacture of cathodic protection rectifiers.

THE DIFFERENCE BETWEEN THIS and THIS





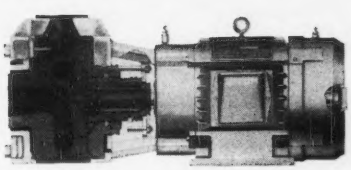
is a corrosion-resistant
"KARBATE" IMPERVIOUS GRAPHITE PUMP!

Why? Because "Karbate" impervious graphite offers total corrosion resistance. It does not rely on a coating or an easily abraded passive or protective film. This absence of corrosion wear in "Karbate" impervious graphite pumps provides continuous, maximum operating efficiency in all recommended applications.

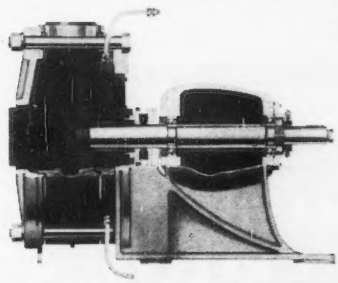
As these facts show, few other pumps — regardless of price — can handle as wide a variety of corrosives as "Karbate" impervious graphite pumps. They are resistant to: *mineral acids* such as *hydrochloric, sulfuric, phosphoric; acid combinations* such as *nitric-hydrofluoric, phosphoric-sulfuric; chlorinated hydrocarbons; alkalies; and organic* and *inorganic compounds of all types.* In practically all of these corrosives, changes in temperature and concentration will not affect the corrosion resistance of "Karbate" impervious graphite.

A "Karbate" pump user comments, "We installed these pumps because they could handle our present variety of solutions and would resist any additional corrosive effects of future changes in these solutions".

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MEN in the NEWS

Donald G. Sturges has been promoted to director of research and development at the Carborundum Company, Niagara Falls, N. Y.

•

Robert G. Vance has been named chief development engineer of Elwell-Parker Electric Company, 4205 St. Clair Ave., Cleveland 3, Ohio.

•

Allyn E. Webb, NACE member and former executive vice president of Barber-Webb Company, Inc., Los Angeles, Cal., has established a consulting service for corrosion problems in industry.

•

Carl H. Samans, NACE member and director of the engineering materials division of the research and development department of Standard Oil Company (Indiana), has been elected chairman of the Chicago section of the American Institute of Mining, Metallurgical and Petroleum Engineers.

•

Douglas F. Williams has been promoted and reassigned to cover southern New Jersey and eastern Pennsylvania for the A. M. Byers Company.

•

Dean E. Ley, formerly with Eastman Kodak Co., has joined the rubber research department of the B. F. Goodrich Company Research Center, Brecksville, Ohio.

•

W. Douglas Cunningham has been appointed district sales manager for Kelite Corporation's Dallas, Texas, District of the Western Sales Region.

•

Otto L. Forchheimer has been named manager of the Chemistry Division of Trionics Corporation, Madison, Wis.

•

John W. Anthony, NACE member, has been named manager of Corrosion Rectifying Company's newly opened North Texas Division.

•

Fred P. Baughman has joined Taylor Fibre Co., Norristown, Pa., as a group leader in the Technical Department.

•

John Boyd, editor of the American Society of Lubrication Engineers' monthly journal, will be given the society's 1960 National Award at the annual banquet, April 20 in Cincinnati, Ohio.

•

Pierre Carpenter, an NACE member, has been promoted to industrial branch manager at Napko Corporation's Houston office. **Joe W. Redden,** chairman of the NACE New Orleans Section, has been appointed industrial branch manager for Napko's New Orleans area.

•

J. Howard Dunn has been named manager of Aluminum Company of America's process development laboratories at New Kensington, Pa.

•

NACE member **Earl A. Erich** has been elected vice president of Tube Turns Plastics, Inc., Louisville, Ky.

•

A. E. Garihan will be manager of Alloy Steel Products Company's new office in Baton Rouge, La.

•

Lyle B. Hoskins has been appointed manager of manufacturing for Carborundum Metals Company's Akron, N. Y., and Parkersburg, W. Va., plants. Reporting to Mr. Hoskins will be **George L. Chapman, Herman H. Brunotte, Robert G. Warrick and Denis A. Kitchen.**

•

George P. Kern, an NACE member, has been appointed to a position in the planning division of the Special Products Marketing Department of Garlock Packing Co., Camden, N. J.

•

Milton D. King, Jr., has been assigned as field engineer in the northern Rocky Mountain oil field area for Hagan Chemicals & Controls, Inc., Pittsburgh, Pa. His headquarters will be in Casper, Wy.

•

Raphael J. Larko has been appointed a regional PVC plastic sales engineer for A. M. Byers Company's Pittsburgh and Chicago sales territories.

•

T. Kenneth McGuire has been named a vice president of the DeVilbiss Company, Toledo, Ohio.

•

John B. McKenna has been named product engineer in charge of chemical feeding equipment for prevention of scale and corrosion at Hagan Chemicals & Controls, Inc., Pittsburgh, Pa.

•

H. E. McNeill has been appointed manager of Dowell's new sales organization that will handle specialty products of the company. Headquarters will be at the company's general offices in Tulsa, Okla.

•

Jean F. Malone has been appointed sales development manager of plastic materials for B. F. Goodrich Chemical Co., Cleveland 15, Ohio.

•

Robert E. Ohlson, NACE member, has been appointed representative for Alloy Steel Products Company. His office will be at 11001 Manchester, St. Louis 22, Mo.

•

Ewell E. Mitchell has been appointed manager of the Goodrich Gulf Chemicals, Inc., plant at Institute, W. Va.

•

H. Grady Rogers, Jr., has been appointed a field service engineer for A. M. Byers Co., Pittsburgh, Pa.

•

NACE member **Richard R. Rue** is now plant manager of Insul-Mastic, Inc., Summit, Ill., a subsidiary of Pittsburgh Coke & Chemical Company.

•

Robert W. Taylor has been transferred to Kansas City, Mo., as technical sales representative for the Metalworking Chemicals Division of Amchem Products, Inc.

•

Ralph A. Schaefer has been named director of Buntin Brass and Bronze Company's enlarged department of engineering and research.

•

John A. Wheeler, Princton University nuclear physics professor, has accepted appointment to the Board of Trustees of Battelle Memorial Institute.

•

Hiram Paul Julien has been appointed manager of advance studies department in the technical branch of the Carborundum Company's Bonded Abrasives Division.

•

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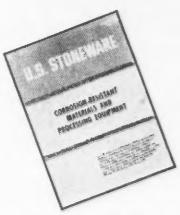
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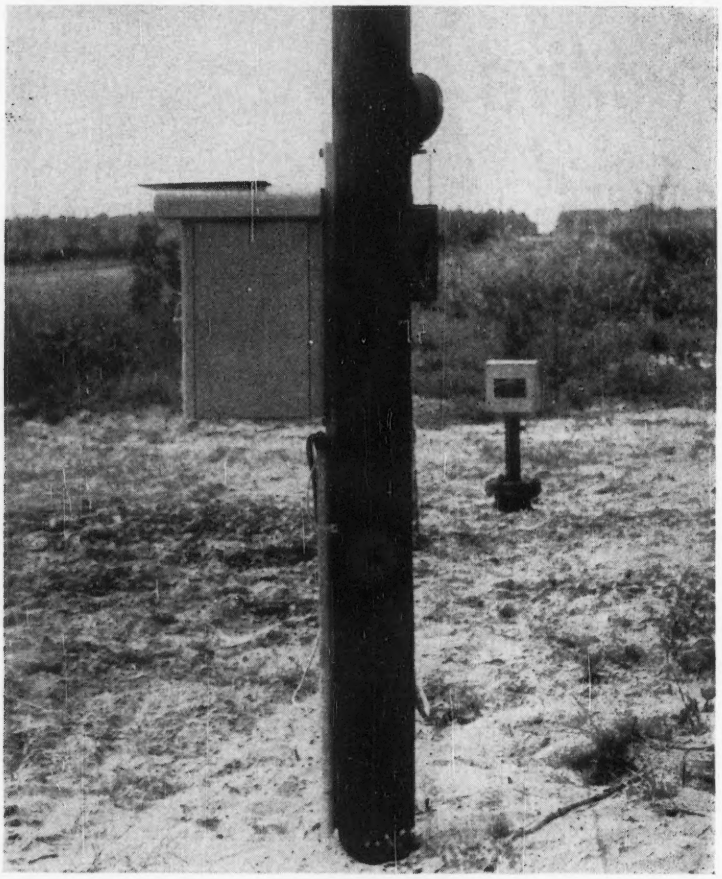
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CORROSION ABSTRACTS

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1. GENERAL

1.2 Importance

1.2.2

Economics of Corrosion. H. Cole. New Scientist, **2,** No. 49, 27-28 (1957).

The economics of the different methods of corrosion prevention are briefly discussed.—RPI. 15758

1.2.5, 3.5.8, 8.9.1

The Safety of Aircraft Structures. A. M. FREUDENTHAL. U. S. Wright Air Development Center. U. S. Air Force, July, 1957, 43 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington, D. C. (Order PB 131382).

Methods of analysis of structural safety are proposed which permit the quantitative evaluation of rational safety factors to be used in the design of airframes for a predetermined risk considered "acceptable" on the basis of expected service life of the aircraft or fleet of aircraft. They also provide a criterion of "fatigue sensitivity" of a design based on certain assumptions concerning load-factors and material performance. The concept of structural safety is analyzed on the basis of its relation to the probability of structural failure. The difference in the approach to the concept of safety for ultimate strength and for fatigue is discussed in the light of recent developments in fatigue research, particularly the results of fatigue tests under random loading. Methods of safety analysis for both conditions are proposed.—OTS. 14947

1.3 Reviews

1.3, 2.3.2, 5.3.4

A Survey of Polish Publications on Accelerated Corrosion Tests of Electroplated Coatings to Determine Their Resistance to Corrosion. (In German.) Tadeusz Biestek. Werkstoffe u. Korrosion, **9,** No. 5, 271-273 (1958) May.

Discussion of several investigations on accelerated testing methods begun in 1953. Electroplated coatings of zinc, cadmium and tin were tested. Thicknesses ranged 1-30 μ. Tests included salt spray tests, immersion tests in distilled water and in ammonium chloride solutions and artificial industrial atmosphere tests. Special research work dealt with accelerated tests of phosphate coatings on steel. Results are discussed and literature references cited. Investigations under way concern chromium, nickel, bright nickel and copper-nickel-chromium coatings.—INCO. 16051

1.3, 3.5.3

Cavitation Erosion—A Review of Present Knowledge. June 1957. D. J. GODFREY. Brit. Ministry of Supply, S & T Memo. No. 17/57, September, 1957, 16 pp.

Cavitation erosion is a type of damage, characterized by pitting attack, which may occur in hydraulic systems having extreme conditions of turbulence or high speed liquid flow. It has been explained for many years as physical damage consequent upon "hammer blows" or shock waves following collapse of vacuum or vapor-filled cavities which may form within liquids when local regions of low pressure arise. Of late it has appeared probable that forces other than of a purely hydromechanical nature may be involved. Thus, the part played by corrosion was found to be important, especially at low cavitation intensities, and cathodic protection was used with success to reduce cavitation erosion. Effects of entrained and dissolved air in cavitation erosion are discussed and dissimilarities and resemblances between impringement attack and cavitation erosion are reviewed. Properties of materials which affect resistance to cavitation erosion, particularly surface hardness and corrosion fatigue resistance, are considered and materials resistant to cavitation erosion are listed. Properties of nickel-manganese steel and nickel-chromium-molybdenum steel are given. Tables, 51 references.—INCO. 15289

1.4 Bibliographies and Indexes

1.4

Heat Transfer and Fluid Flow: A Bibliography of Selected Report Literature. James M. Jacobs, compiler. Technical Information Service Extension. U. S. Atomic Energy Commission, Pubn. TID-3305 (Suppl. 1), June 1958, 435 pp. Available from Office of Technical Services, Washington, D. C.

A total of 2519 annotated references to the unclassified report literature is presented. Subjects covered under heat transfer and fluid flow include radio-induced heating, boiling, boilers, evaporators, pumps, heat exchangers, hydrodynamics, coolants and their properties, thermal and flow instrumentation, high-temperature materials, thermal properties of materials and thermal insulation. Subjects covered less completely include thermodynamics, aerodynamics, high-temperature corrosion, corrosion specific to heat transfer systems, erosion, mass transfer, corrosion film formation and effects, coolant processing and radioactivity, radiation effects of heat transfer materials, and pertinent data of thermonuclear processes. Subject, report number and author indexes are given. (auth)—NSA. 16581

1.4, 4.6.2, 3.2.3, 3.6.2, 3.6.6

Stress, Crevice, Galvanic and Uniform Corrosion of Austenitic Stainless Steels and Carbon Steel in High Temperature Boiler Feed Water: A Literature Review. R. B. Richman. General Electric Co. Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-54879, Feb. 6, 1958, 12 pp. Available from Office of Technical Services, Washington, D. C.

A bibliography of 89 references on stress, crevice, galvanic and uniform corrosion of stainless and carbon steel in high temperature boiler water is presented.—NSA. 17584

1.4, 8.4.5

Materials for Use in Nuclear Reactors. Information Bibliography. B. Yates. United Kingdom Atomic Energy Authority, 1958, 56 pp. Available from British Information Service, 45 Rockefeller Plaza, New York, N. Y.

A bibliography of the choice and properties of reactor materials, excluding fuel materials except where coincidental, to be used in reactors and reactor environments is presented. 366 references.—NSA. 17582

Abstracts in This Section are selected from among those supplied to subscribers to the NACE Abstract Punch Card Service. Persons who are interested in reviewing all available abstracts should write to NACE for information on this service.

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NACE will NOT accept orders for photoprint or microfilm copies of material not published by the association.

1.7 Organized Studies of Corrosion

1.7.1

First European Congress on Corrosion. (In French.) J. L. Rabate. Tech. l'Eau (Brussels), No. 123, 47-49 (1957) March 15.

Summary of main points of reports presented to Congress (Paris, 11/18-12/3/56) on the following general themes: Methods of combating corrosion; corrosion and protection of manufactured objects; economic aspects of corrosion.—MR. 15404

1.7.1

General Report of the Congress. (In French.) M. P. Lacombe. Chim. Ind., 77, 29-32 (1957) April.

Summarizes the main patterns of thought revealed by 24 papers presented at the European Corrosion Congress in November, 1956. Present-day tendencies of scientific research into corrosion, industrial applications, modern processes of protection. Fundamental research conducted into corrosion, methods of study, methods of testing, methods of protection, problems of atmospheric corrosion and light alloys.—MR. 16215

1.7.1, 3.2.2, 3.7.4, 6.2.4

Organization and Tasks of the Swedish High-Temperature Materials Committee. S. Arwidson. Arch. Eisenhüttenw., **28,** 673-674 (1957) Nov.

Constitution and terms of reference of this committee and its four subcommittees (Testing Equipment and Methods, Embrittlement Phenomena, Effects of Structure and Grain Size on Creep Strength, and Effects of Alternating Loading and Temperature). Present state of work in hand includes: studies of embrittlement in low-alloy nickel-chromium-molybdenum steels and 20/20 chromium-nickel steels, assessment of notch-impact test in relation to thermal embrittlement, and effects of ferrite grain size and carbide distribution on creep behavior of chromium-molybdenum steels and of austenite grain size in 16/13 chromium-nickel steels. Elevated-temperature fatigue tests on low-alloy chromium-molybdenum steels are referred to.—INCO. 15193

1.7.1, 4.6.1

Corrosion in the Research Program at Cebedeau. (In French.) E. Leclerc. Corrosion et Anticorrosion, **5,** 185-188 (1957) June.

Research currently in progress and projected at the Centre Belge d'Etude et de Documentation des Eaux (Cebedeau). Work is concerned with potable waters, soft waters and waters for boilers; an experimental high-pressure boiler is being built. 9 references.—MR. 15341

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41a

3. CHARACTERISTIC CORROSION PHENOMENA

3.2 Forms

3.2.2, 6.4.2, 2.3.2, 2.2.3

Intergranular Corrosion of Duralumin: Correlation of Laboratory and Service Behavior. G. J. Schafer, T. Marshall and H. J. Todd. J. Applied Chem., **9**, Pt. 1, 38-43 (1959) Jan.

An accelerated test for susceptibility to intergranular corrosion was applied to a number of aluminum-copper alloys whose service corrosion behavior is known. Specimens were collected from roofs, old aircraft and other structures and a number of long-term corrosion test panels were examined. All specimens (core alloys only, in case of clad material) were analyzed spectrographically (nickel content of 0.01-0.1). In all cases, where intergranular corrosion occurred in service, samples showed same type of corrosion in acid chloride test solution. There was good correlation between type of attack in test solution and K value (ml./cm.^{3}/min.^{2}) relating volume of gas evolved in test and square of time of effective attack. Factors which affect service performance are discussed and include cladding, ageing effects, grain size and environment. Table, graph, 6 references.—INCO. 17816

3.2.2, 3.5.9, 6.2.3

Effects of the Duration of the Load and the Maintenance of Ambient Temperature on Hydrogen Embrittlement in Steel. (In French.) Jacques Plusquellec, Pierre Azou and Paul Bastien. Compt. rend., **246**, 3454-3457 (1958) June 23.

The effect of the duration of loading and the duration of maintenance at ambient conditions on embrittlement of extra-mild steels by electrolytically introduced hydrogen were studied. The details of the loading are described. (auth).—NSA. 17562

3.2.2, 8.4.3, 5.7.2, 3.4.7

You Can Stop Hydrogen Attack. W. A. Bonner. Petroleum Refiner, **37**, No. 7, 111-114 (1958) July.

Injection of air into gas streams with controlled pH has been used successfully to prevent hydrogen attack in locations where cyanide concentration is relatively low. Preformed polysulfide solution may be used in conjunction with air injection when cyanide concentration is sufficient to prevent desired protection with air injection. Causes of hydrogen attack are reviewed and conditions promoting attack considered. Reactions of the process are discussed. Water must be held at pH of 7.8-8.3; air injection causes corrosion at pH less than 7.8 and solids may deposit at pH above 8.5. Economics in use of Shell's process are considered.—INCO. 18013

3.2.2, 6.2.3, 6.2.2

Hydrogen Embrittlement and Internal Friction of Carbon Steel. (In Japanese.) Genjiro Mima and Michio Mizuta. Tetsu-to-Hagane, **43**, No. 2, 132-137 (1957) February.

Experiments with hypo-eutectoid carbon steels (C 0.07-0.68%) including pure iron and eutectoid steel (C 0.85%); the authors measured amounts of evolved hydrogen, changes in internal friction (as a structure-sensitive quantity) and mechanical properties (as a measure of actual embrittlement) during the evolution (at room temperature) of electrically-charged hydrogen; investigation of the relationships among these different factors.

Results: Microscopically speaking, diffusible hydrogen (at room temperature) was absorbed more in free ferrite than ferrite in pearlite, but macroscopically existed in uniform distribution of concentration. Hydrogen evolved according to the diffusion process described in the Fick's formula, and the diffusion constant was found to be $D = 3.7 \times 10^{-5}$ cm^2/min. (at room temperature).

Ageing was found to cause a decrease of the elongation and reduction of area in tensile test, a decrease of the impact value, and an increase of the internal friction.

Though the recovery of ductility in the tensile test runs parallel to hydrogen evolution, the recovery of internal friction was more rapid than the former; and the recovery of impact value was far more rapid. 17971

3.6 Electrochemical Effects

3.6 8, 6.4.4

Hydrogen Overvoltage on Magnesium. (In Russian.) B. N. Kabanov and E. V. Barelko. J. Phys. Chem., USSR (Zhur. Fiz. Khim.), **31**, No. 11, 2501-2506 (1957).

Hydrogen overvoltage (η) on magnesium in potassium hydroxide solution is independent of potassium hydroxide concentration from 0.1 to 2 N. On a mechanically cleaned magnesium surface η is approximately the same as on an ideally clean surface, i.e. 1.4 ± 0.1V. On a surface anodized at a current density of 2 x 10^{-2} amp/cm^2, η is 0.5V higher and the η/log i curve exhibits a sharp break. Large concentrations of chlorine ions lower η in dilute potassium hydroxide solution. This can be due to displacement of oxygen by chlorine ions by adsorption. Measurement of the capacitance of the double layer shows that neither oxidation nor additions of chlorine ions affect the capacitance and therefore oxidation and adsorption of chlorine ions does not cause appearance or disappearance of a separate oxide phase on the surface.—MA. 17858

3.6.5, 2.3.5, 3.2.2, 6.2.3, 6.2.5

Determination of Causes of Corrosion by Electrochemical Measurements. (In German.) H. Schmecken. Werkstoffe u. Korrosion, **9**, 690-693 (1958) Nov.

Semilogarithmic plots of current density versus voltage disclose nature and magnitude of corrosion currents. In mild steel, corrosion sensitivity depends strongly on chemical composition, showing marked improvement in high-carbon steels. A 18% chromium, 10.5% nickel stainless steel in commercial phosphoric acid bath generates static potentials which cause pitting of contaminated surfaces.—RML. 17766

3.6.6, 8.1.4, 5.4.5

Investigations of Corrosion in Water Mains and Methods of Protection. (In German.) C. Tschappat. Werkstoffe u. Korrosion, **9**, No. 10, 613-623 (1958) October.

Rusting of pipes is discussed first in terms of general galvanic theory. A summary of the behavior of different metal parts of pipelines is given for percolating water and temperature and aeration fluctuations. Some protective measures are explained, and a paint coating claimed to give durable protection is described.—ZDA. 17589

3.6.6, 3.6.8

Relation Between the Contact-Corrosion Rate and Polarization Characteristics of Multiple Electrodes. (In Japanese.) Namio Ohtani. J. Japan Inst. Metals (Nippon Kinzoku Gakkai Si), **21**, No. 11, 674-677 (1957).

The galvanic corrosion-current of zinc in contact with iron, is analyzed graphically and represented as a function of the ratio of the anodic and cathodic polarizations, the cathode area, the heat of activation of the cathode metal in the hydrogen electrode reaction and the ion concentration of the cathode metal in solution.—MA. 17953

3.6.6, 5.4.5, 8.1.2

Corrosion of Metals in Buildings. J. I. Lewis. Paint J., **7**, No. 53, 422-423, 458 (1957).

The mechanism of corrosion of metals and some preventive measures are outlined. The dangers of bi-metallic corrosion are stated. Copper should never be used in contact with either galvanized steel or aluminum unless the joints are adequately protected by at least two coats of a good-quality bituminous paint. —ZDA. 17912

3.6.8, 3.7.2, 3.8.4, 6.3.20

Role of Hydrogen in the High-Temperature Corrosion of Zirconium and Its Alloys. Pt. I. The Effect of Cathodic Polarization on Corrosion in Water at 325 C. J. N. Wanklyn and B. E. Hopkinson. J. Applied Chem., **8**, No. 8, 496-504 (1958).

The study was carried out on binary zirconium alloys and Zircaloy-2, to ascertain the effects of alloying additions and the cathodic discharge of hydrogen. In some cases cathodic polarization increased the uptake of hydrogen and reduced the protective character of the oxide films, but no systematic relationship between the latter two factors was observed. Films formed in a previous period of unpolarized corrosion were damaged when polarized at 325 C and at room temperature. It is suggested that hydrogen exerts its harmful influence by passing through the oxide and into the metal, rather than by modifying the film-growth process, and that beneficial alloying additions act by inhibition of the effects of hydrogen on the oxide.—MA. 17819

3.7 Metallurgical Effects

3.7.2, 6.4.4

Influences of Impurities on Some Properties of High Purity Magnesium. Pt. III. The Removing Effects for Impurities by Zirconium. (In Japanese.) Riei Ichikawa. Light Metals, No. 25, 65-68 (1957) July.

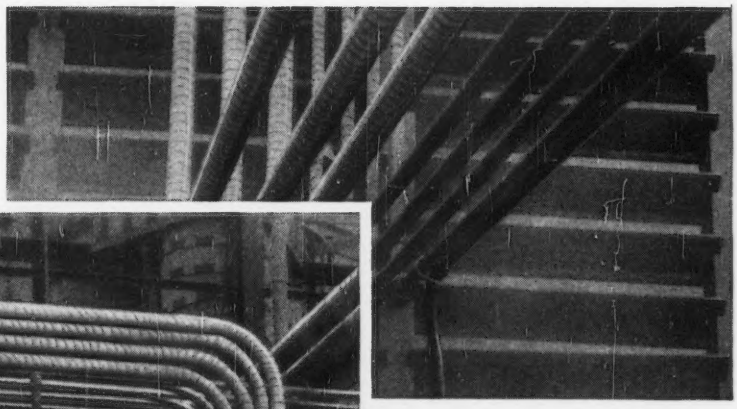
The refining effects for molten magnesium contajning iron, copper, manganese, silicon and aluminum as impurities by the addition of zirconium were investigated by chemical analysis and corrosion test. Elements such as iron, manganese, silicon and aluminum were removed easily in the written order, the first being the easiest. Hence iron in magnesium was removed nearly perfectly, manganese and silicon in magnesium were removed substantially, but copper in magnesium was not removed by the addition of zirconium. (auth).—ALL. 17949

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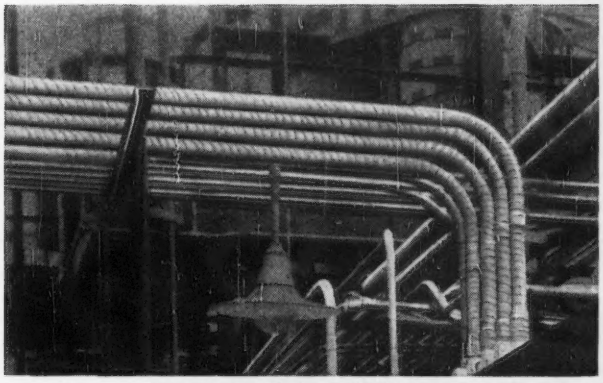
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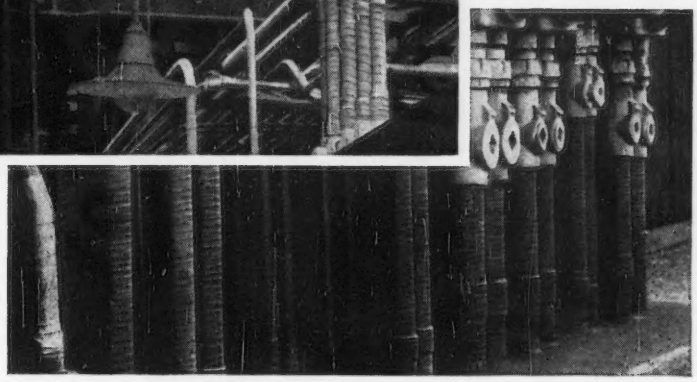
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3.7.3, 6.3.10

Influence of Heat-Treatment on Some Properties of Corrosion-Resistant Nickel-Molybdenum (-Iron) Alloys. (In German.) Karl Bungardt and Hans-Hermann Weigand. Z. Metallkunde, **50,** No. 1, 11-18 (1959).

Alloys containing nickel 56.8 + molybdenum 21.1, nickel 63.2 + molybdenum 29.4, nickel 58.6 + molybdenum 16.9 + chromium 17.5, and nickel 46 + molybdenum 6 + chromium 21.1 + Ta(Nb) 2.2%, in all cases the balance being iron, with small amounts of carbon, manganese and silicon, were studied. Solution-treatment was 15 min at 1175-1225 C, followed by quenching in water and precipitation-hardening at 500-1100 C for up to 100 hr. Hardness increases most during the first 25 hr of ageing, increases with increasing temperatures between 700 and 900 C and decreases at high temperature. The P.S. and U.T.S. attain maximum and the elongation and reduction in area attain minimum on ageing at 800-900 C. Resistance to boiling 10 and 21% hydrochloric acid and 20% sulfuric acid + 2% nitric acid decreases with increase in precipitation-hardening, and can be related to the increasing amounts of the molybdenum (and, when present, chromium) hardening phase being precipitated and coagulating at grain boundaries; corrosion-resistance increases only when the ageing temperature and time are great enough to produce molybdenum diffusion, therefore, from the grains to the impoverished regions of the grain boundaries and thereby restore, to some extent, equilibrium conditions.—MA. 17786



3.7.3, 6.3.15, 3.7.4

Practical Heat-Treatment for Titanium Alloys. P. D. Frost. Metal Treatment, **24,** No. 143, 307-312 (1957).

Difficulties are considered. The effect of alloying elements and cooling rates on the stability of the β phase and its tendency to transform to brittle ω phase are outlined. Overheating in the β region can lead to excessive grain-growth and consequent poor ductility. Oxidation during heating and distortion when quenching are also considered and remedies suggested. 10 references.—MA. 17955

3.7.4, 3.7.2, 6.2.5

Phases Present in Chromium-Nickel Austenitic Steels Containing Titanium. (In Russian.) V. I. Prosvirin. Metalloved. I. Obrabotka Metallov., No. 10, 2-5 (1958) October. Translation Available from: Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, California.

Correlations regarding the formation of titanium-containing phases in austenitic steels. Composition ranges studied: 0.06-0.10% carbon, 13.4-16.1 chromium, 15.6-25.3 nickel, 0.68-2.66 titanium. Composition of secondary phases after quenching; effect of quenching temperature on amount of titanium in secondary phases. Variation of amount of electrolytic residue with aging temperature and corresponding variation of percentage of titanium. Changes in content of the various components in the phases in weight of the electrolytic residue and lattice parameter with aging temperature. 6 figures.—HB. 17553

3.8 Miscellaneous Principles

3.8.4, 3.7.4, 6.2.2

Oxide Nucleation and the Substructure of Iron. E. A. Gulbransen and K. F. Andrew. Westinghouse Electric Corp. J. Electrochem. Soc., **106,** No. 6, 511-515 (1959) June.

Electron optical studies were made of the initial stage of reaction of oxygen with pure iron. Oxidation occurred in a discontinuous manner with the oxide structure orienting to the metal grain. The sites for oxide nucleation depend on the initial pretreatment atmosphere. Thus, a vacuum annealing treatment showed a random arrangement of nucleation sites, while a hydrogen annealing treatment showed a highly ordered arrangement of nucleation sites.

Since iron was embrittled by annealing in dry and moist hydrogen and not by vacuum annealing, the nucleation sites probably showed the substructure of iron responsible for embrittlement. 17803

3.8.4, 6.2.2, 3.5.9, 3.7.4, 2.3.9

Oxidation Studies on the Iron-Chromium-Aluminum Heater Alloys. Earl A. Gulbransen and Kenneth F. Andrew. Westinghouse Electric Corp. J. Electrochem. Soc., **106,** No. 4, 294-305 (1959) April.

The oxidation behavior of three iron-chromium-aluminum alloys was studied using sensitive weight gain techniques. In addition, x-ray diffraction methods were used to determine the crystal structure and composition of the oxide films. The adhesion characteristics of the oxide film to the metal were tested by introducing strains into the metal oxide system during oxidation.

Rate studies show a change in rate for the three alloys near 900 C. Between 900 to 1050 C the rate of oxidation remained nearly constant; above 1050 C it increased again. X-ray diffraction studies suggest that a crystal structure transformation was occurring in the oxide. At 1050 C, α-aluminum oxide was the main oxide in the scale.

The parabolic rate law was used to interpret the rate data. Deviations occurred during the initial reaction period as a result of crystal structure transformations in the film. Heats of activation of 76 to 77 kcal/mole were calculated for the range 700-900 C.

Strain oxidation studies showed the oxide scale on the high aluminum alloy to be less susceptible to damage than the lower aluminum content alloys. These studies were correlated directly with practical performance tests.

Thermodynamic analyses of the several types of solid-solid and solid-gas reactions were used to interpret the rate and structure studies. Preliminary design principles were proposed for heat resistant alloys. 17773

3.8.4, 6.3.17, 4.6.2, 3.5.9

Kinetics of the Uranium-Steam Reaction. B. E. Hopkinson. Atomic Energy Res. Establishment, Harwell, England. J. Electrochem Soc., **106,** 102-106 (1959) Feb.

Reaction rates of unalloyed uranium with superheated steam at 160 to 1400 C and 1 atm. pressure have been determined by a thermogravimetric method. Up to 880 C the reaction follows a linear rate law with maximum rates at approximately 300 and 750 C. Above 880 C a parabolic rate law applies for the first 60 to 120 minutes, after which corrosion is linear; the oxide formed in the initial period gives protection to the underlying metal, and the subsequent linear rate is as low as that at about 200 C. X-ray diffraction of the reaction product indicated that uranium dioxide was formed over almost the whole temperature range. (auth).—NSA. 17720

3.8.4, 6.3.20, 3.4.9, 3.5.9

Oxidation of Zirconium at High Temperatures and the Structure of the Primary Oxide Layers. (In Russian.) E. S. Sarkisov, N. T. Chebotarev, et al. Atomnaya Energiya, **5,** No. 5, 550-553 (1958).

Several stages of zirconium oxidation in oxygen and water vapor were observed at 150 to 800 C. A fine layer of cubic structure and high passivity appears in the first stage, followed by a texturized monoclinic modification right under the cubic structure. In the last stage the black layer of cubic and monoclinic structure is replaced by a white film, followed by a sharp increase in the oxidation rate. The high corrosion resistance of the black zirconium film is related to the texturized solid solution of zirconium in zirconium dioxide. (auth)—NSA. 17754

3.8.4, 3.7.4, 6.3.6

Thermal Oxidation of Copper (111) and (100) Faces. Y. N. Trehan and A. Goswami. Trans. Faraday Soc., **54,** 1703-1708 (1958) Nov.

Oxidation was carried out in air in the temperature range 120-350 C. The initial oxide layer formed consisted of cuprous oxide having an amorphous or a fine-grained structure. Increase in film thickness with temperature and/or time of oxidation resulted in the formation of crystalline cuprous oxide grown epitaxially with a two-degree (111) orienta-

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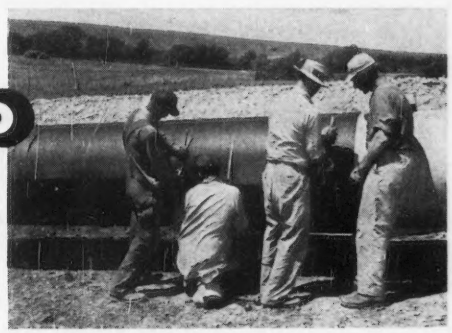
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tion on both the faces. With further increase in temperature or thickness, mixed two-degree (111) + (100) orientated cuprous oxide was formed presumably as a result of multiple twinning and finally the top layers consisted of randomly disposed or one-degree orientated cuprous oxide. 1 plate.—BTR.
17627

3.8.3, 6.3.10
On Flade Potential of Passive Nickel. (In Japanese.) G. Okamoto, T. Takaishi and N. Sato. Paper before Electrochem. Soc. Japan, June, 1958. J. Electrochem. Soc. Japan, **26,** No. 12, 615-618 (1958) Dec.

Through Flade potential a surface behavior of passive nickel was investigated. The behavior of passive nickel was a little different from that of passive iron. The difference between measured Flade potential and oxide formed potential of passive nickel was similar to that of passive iron. But an approximately calculated value of chemisorbed oxygen formed potential of passive nickel was much smaller that that of its Flade potential. This fact does not agree with Uhlig's chemisorption hypothesis which was deduced from the study of passive iron. As significantly the passive film is thin, the Flade potential was calculated by the surface free energy of the oxidized film on nickel. The surface free energy depends on the thickness of the film. Assuming that nickel monoxide film being about 70 A in thickness, the Flade potential was calculated as an equilibrium potential from the reaction $NiO + 2H^+ + 2\theta \rightleftharpoons Ni + H_2O$.—INCO.
17527

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3.8.3, 6.2.2
Passivity of Iron in Hot Alkaline Solution. (In Russian.) A. M. Sukhotin and K. M. Kartashova. J. Phys. Chem., USSR (Zhur. Phiz. Khim.), **32,** 2068-2072 (1958) September.

The substance of the passivating film on iron in a 10 normal sodium hydroxide solution of 80 C, as in acid solutions, is magnetic iron oxide. In system studied, magnetic film may attain considerable thickness but the passivating action is connected only with a very thin layer directly adhering to the metal surface.—MR.
17850

3.8.3, 5.9.4
Structure of Passivating Films. (In German.) Kurt Huber. Z. Elektrochem., **62,** No. 6/7, 675-683 (1958).

The structure of passive films on zinc, aluminum, tin and silver was studied by electron-diffraction and replica techniques. The structure of passive films on zinc varies with the conditions of formation. For anodic films the same structure occurs in completely different chemical systems. Primary anodic oxide films can transform by reaction with the electrolyte forming special structures. 43 references.—MA.
18014

4. CORROSIVE ENVIRONMENTS

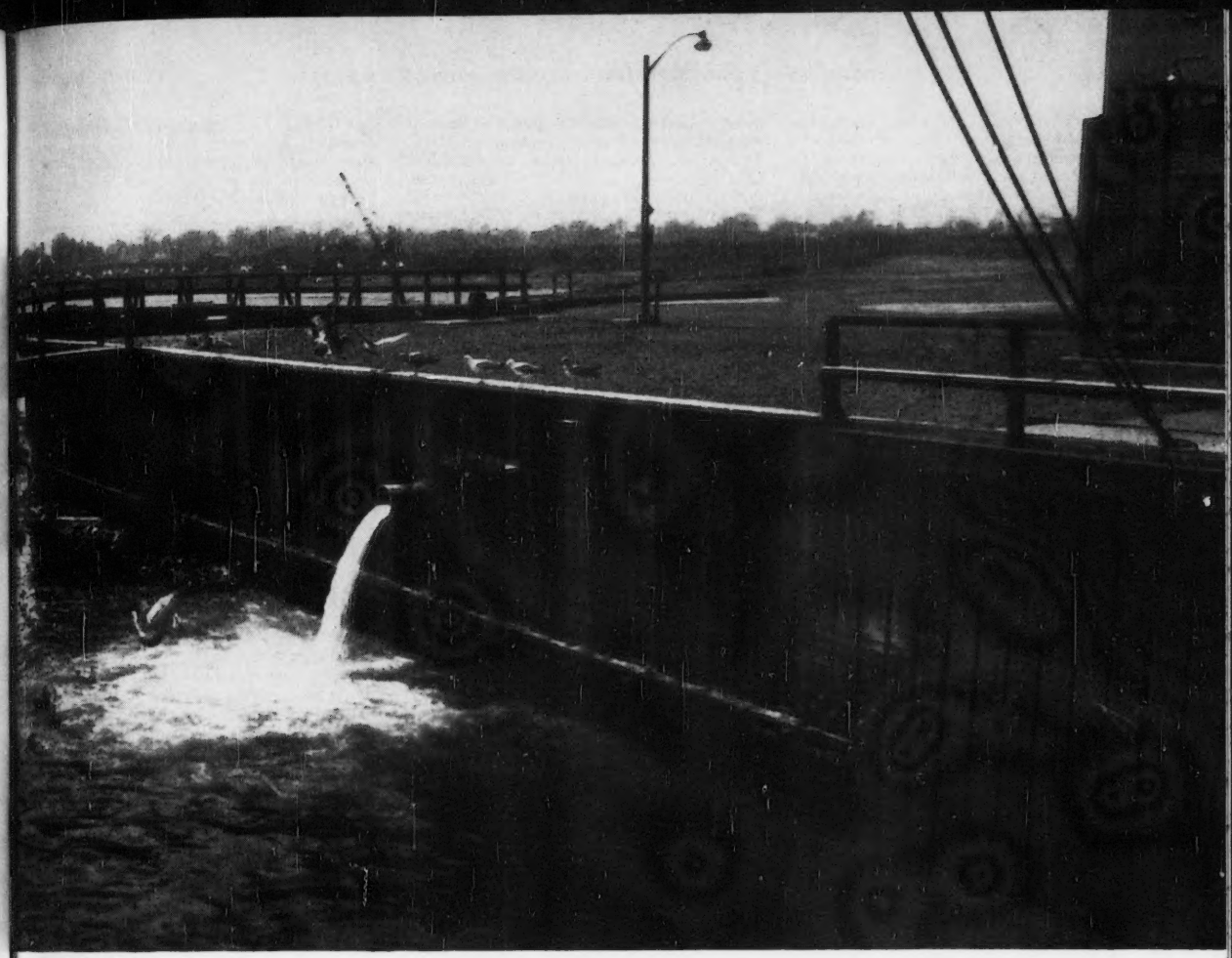
4.2 Atmospheric

4.2.1, 3.5.8, 3.2.2
Effect of Atmospheric Corrosion on Metal Fatigue. (In English.) N. J. Wadsworth and J. Hutchings. Philosophical Magazine, **3,** No. 34, 1154-1166 (1958) Oct.

Polycrystalline specimens of copper, aluminum and gold were fatigued in reversed bend in various pressures of air and in water vapor and inert gases. The ratio of life in atmosphere of air to that in vacuum (10^{-5}mm mercury) at lives of about 10^6 cycles was 1:20 for copper, 1:5 for aluminum and about 1:1 for gold. Some specimens were examined at intervals during test. Small cracks a few microns deep formed in the few hundreths of the life in air and after the same number of cycles in vacuum. The effect of air was to speed the propagation of cracks in copper and aluminum but not in gold. Electron micrographs showed that extrusions and intrusions can occur close to each other in the same slip band. (auth).—ALL.
17671

4.2.3, 7.1, 7.3
Rocket Exhaust Corrosion Simulator. F. B. Cramer. Rocketdyne, North American Aviation, Inc. U. S. Wright Air Development Center, U. S. Air Force, August, 1957, 138 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 151273).

Techniques for studying the corrosive effects of rocket exhaust gases upon various metal and paint-coated surfaces were devised and designs of the items of test equipment and methods of operation are presented. Designs were developed for a device capable of providing uniform exposure of large numbers of specimens to a single firing of a solid propellant charge. One design is for a laboratory device to burn small quantities (one pound) of propellants under very low hazard conditions. Another is for a device capable of uniformly exposing 72 specimens to multiple samples of the hot, high-velocity exhaust gases of



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an air-craft armament rocket. Application of the equipment showed that exposure was uniform from specimen to specimen and that differences could be demonstrated between different surfaces. Evaluation also showed that armament rockets could be compared statistically with propellants burned in a laboratory burner. The devices also permit specimens exposed to rocket exhausts during airflight to be qualitatively compared to the specimens exposed to propellants burned in the laboratory.—OTS. 17855

4.2.3, 3.8.4, 6.6.6

Oxidation of Zirconium Carbide (and Titanium Carbide) in High-Temperature Combustion Gases. W. Watt. Powder Met., No. ½, 227-234 (1958).

Reactions of sintered zirconium and titanium carbides were studied in simulated rocket-combustion gases at surface temperatures of approximately 2250 C. Zirconium carbide is oxidized according to a parabolic law owing to the formation of an adherent oxide film. No oxide film is formed on titanium carbide under the same conditions, and it decreases in weight and dimensions at a relatively rapid rate. (auth)—MA. 17672

4.2.3, 3.4.9, 6.2.3

Resistance of Ferrito-Pearlitic Steels to Corrosion in Water Vapor and in Combustion Products at Temperatures Above 530 C. (In Czech.) Bohumil Prenosil. Hutnické Listy, 13, No. 7, 603-612 (1958).

Growth of protective oxide layer. Effects of water vapor pressure, pre-stress, sodium phosphate contamination, contamination with sodium hydroxide on corrosion penetration. Presence of sulfur dioxide and oxygen in combustion products and their influence on corrosion. Comparison between corrosion resistance in water vapor and in combustion products. 13 references.—MR. 16956

4.5 Soil

4.5.1, 6.2.2

New Methods of Iron Protection in Soils. T. Markovic. Werkstoffe u. Korrosion, **8,** No. 10, 596-602 (1957) October.

Conditions for active and passive behavior of iron are considered. Structure of soil with respect to alterations of capillary and noncapillary porosity plays decisive role. Factors such as water and air in soil, soil resistance and acidity, which influence corrosion processes, are discussed. Ratio of water to air in soil determines the corrosion rate. Every soil has a specific aggressiveness which is determined by water capacity of the soil. Reducing and eliminating corrosion with and without cathodic protection, by addition of inhibitors, and by preparation of artificial aeration cells, is discussed.—INCO. 15367

4.5.1, 5.4.5, 3.3.4

Biological Corrosion in Hydroelectric Works in Africa and the Action of Organic Products in This Corrosion Process P. Colomb. Corrosion et Anti-corrosion, **5,** No. 5, 153-155 (1957); Paint Ind. Res. Inst. Lit. Dig., **8,** No. 2, 40 (1957).

An attempt is made to explain the poor performance of classical bituminous coatings on pipes in the Belgian Congo. In an examination of mud deposits, no bacteria known for corrosive action were found, but the aggressive action of the water could be explained by the presence of amino acids and amino bases from rotting animal and vegetable matter. These were detected by chemical and chromatographic analysis. This could also explain the unexpected resistance of certain paints based on red lead.—RPI. 14615

4.5.3

Corrosion Investigation by Electrical Measurements. D. Hendrickson. Corrosion Technology, **4,** 390-393 (1957) Nov.

Application of soil surface potential method of survey to locating corroding areas and approximating current density of discharge for Mokelumne Aqueduct consisting of 81 miles of parallel bituminous coated and cement mortar coated pipes.—MR. 16186

4.7 Molten Metals and Fused Compounds

4.7

Corrosion Testing of Various Materials in Sodium. Pt. I. R. F. Dudek. Pt. II. K. Mildred Ferguson. Babcock & Wilcox Co. U. S. Atomic Energy Commission Pubn., BW-7020, April 26, 1957, 39 pp. Available from Office of Technical Services, Washington 25, D. C.

These tests were conducted to determine the corrosion resistance of various materials to liquid sodium at a temperature of 1100 F. Four separate tests were conducted using a modified enclosed-rotor pump corrosion apparatus with sodium at 100 F and with the specimens at an absolute velocity of 55 ft/sec and static. The materials tested were a group of 300 and 400 series stainless alloys, low Croloys, carbon steel, nickel alloys, zirconium and Lukens-clad stainless steels. Oxygen was controlled by the use of a diffusion-type



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(2) **Measure pipe-to-soil potentials with the reference half-cell located on the opposite side of other pipe lines?** Again, the answer is "No." Also, in running Log Current-Potential curves on wells, it is important that the half-cell have an unobstructed view of the well under test.

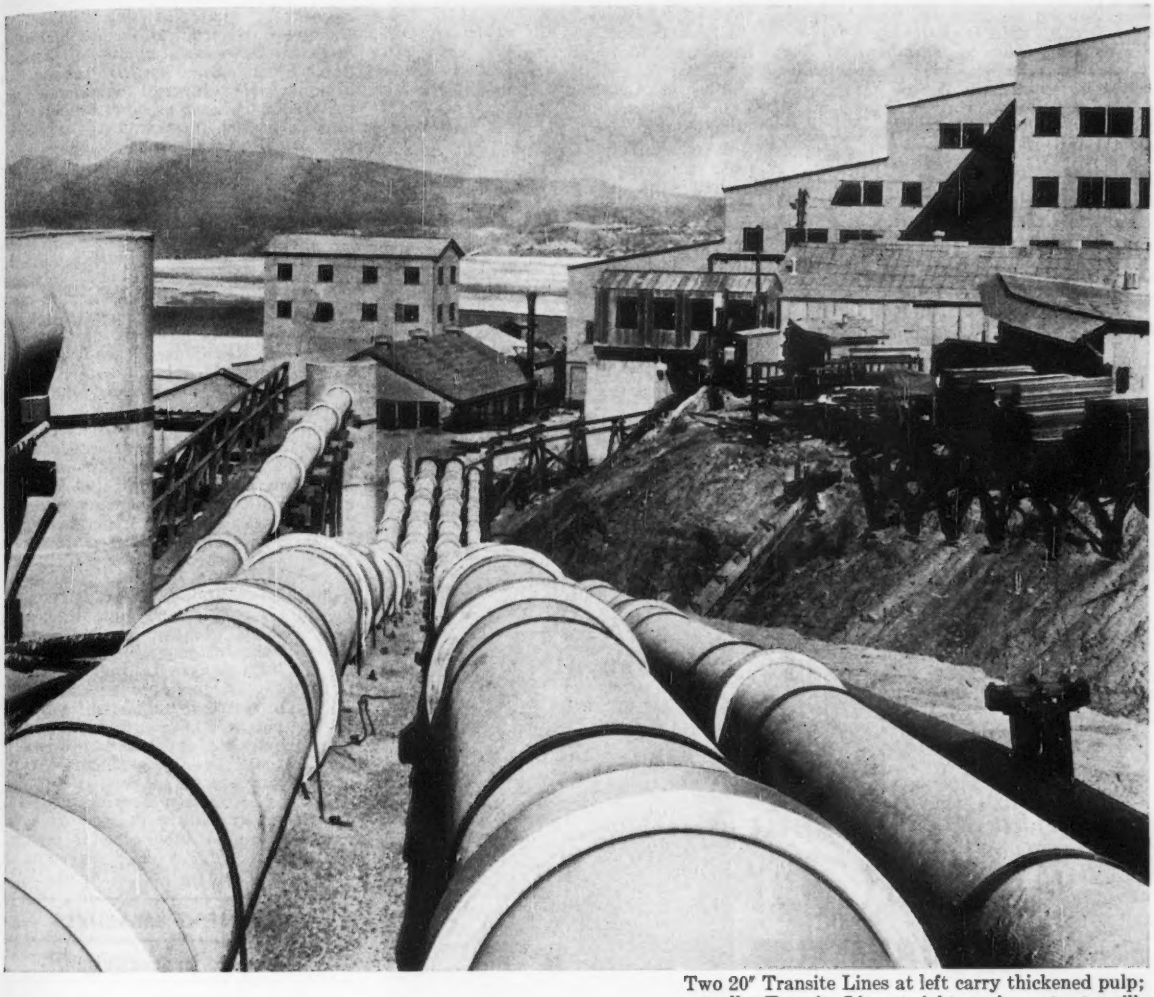
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49a

cold trap for three of the four tests. A flow-through cold trap was used for the fourth test. The measurement of the oxygen content was made both chemically and by the use of a plugging indicator. After test, the specimens were subjected to metallographic investigation. (auth) 17963

4.7, 3.7.4

Mechanism of Corrosion of Metals in Molten Chlorides. (In Russian.) N. D. Tomashov and N. I. Tugarinov. J. Applied Chem. USSR (Zhur. Priklad Khim.), **30**, 1681-1687 (1957) Nov. Translation Available from Consultants Bureau, Inc., 227 West 17th Street, New York 11, N. Y.

Activity and prevention of high-temperature corrosion; 70% of surface corrosion concentrated around grain boundaries. 11 references.—RML. 18004

4.7, 3.5.4, 6.3.10, 8.4.5

Effect of Radiation on Corrosion of Structural Materials by Molten Fluorides. G. W. Keilholtz, J. G. Morgan and W. E. Browning. Oak Ridge Natl. Lab. Nuclear Sci and Eng., **5**, 15-20 (1959) Jan.

A survey of the experimental methods used in testing the radiation stability of molten salts and their corrosion properties is presented. The effects of irradiation on the corrosion of Inconel exposed to fluoride fuel mixtures and on the physical and chemical stability of the fuel mixtures were investigated by irradiating in the MTR capsules filled with static fuel and by operating in-pile forced-circulation loops in the LITR and in the MTR. In the many capsule tests and in the three in-pile loop tests made to date, no major changes have occurred in the fuel mixtures that can be attributed to irradiation, other than normal burn-up of uranium. Metallurgical examination of the Inconel capsules and tubing have likewise shown no changes in corrosion that can be the result of radiation damage. The low corrosion results obtained for the in-pile loops have been confirmed by chemical analyses for corrosion products in the fuel mixtures. (auth)—NSA. 17623

4.7, 8.4.5

Behavior of Fission Products in Molten Fluoride Reactor Fuels. Mark T. Robinson, William A. Brooksbank, Jr., Samuel A. Reynolds, Henry W. Wright and Thomas H. Handley. Oak Ridge National Lab. Nuclear Sci and Eng., **4**, 288-296 (1958) Sept.

Observations are reported on the behavior of several fission product elements in molten sodium fluoride-zirconium fluoride-uranium tetrafluoride fuels, irradiated in capsule experiments, forced-convection in-pile loop experiments and in the Aircraft Reactor Experiment (ARE). The rare gases were observed to escape readily from the fuels in dynamic tests, although in static tests the rate of escape is very low. Ruthenium and niobium deposit on the Inconel walls of the fuel container, probably as metals. Other fission products studied (strontium, zirconium, lanthanum, cerium) appear to remain in the fuel. The results obtained are entirely consistent with theoretical predictions. It is suggested that the observed noble metal deposit may serve to reduce corrosion of metals by molten fluoride fuels. The unsatisfactory nature of Cs^{137} as a fission monitor in such fuels is reported and the use of Zr^{95} as a substitute is discussed. (auth)—NSA. 17585

5. PREVENTIVE MEASURES

5.2 Cathodic Protection

5.2.1, 5.9.4, 5.4.5, 8.9.1

Protection of Light Metal Assemblies in Aircraft Construction. (In French.) Paul Roussetbert. Corrosion et Anticorrosion, **6**, No. 9 272-287 (1958) Sept.

Cathodic protection (cladding), chemical oxidation, anodic oxidation and organic coatings as applied to wing and fuselage assemblies, homogeneous or heterogeneous, either riveted, welded or bolted. Role of each type of protection; principles and description of processes. —MR. 17570

5.2.1, 8.9.3

Certain Aspects of Cathodic Protection. (In German.) K. Thalhofer. Werkstoffe und Korrosion, **9**, No. 8/9, 515-517 (1958) Aug.-Sept.

Construction of modern cathodic protection installations for old pipelines (water, gas) in densely populated as well as in industrial districts is meeting increasing difficulties. The number of buried pipes makes it necessary to extend the protective installations.—RML. 17602

5.2.2, 4.6.11

Sea Water as Industrial-Water; Cathodic Protection from Electrolytic Corrosion. (In Japanese.) T. Otobe. Metals, **28**, 652-658 (1958) Sept.

The screen for an inlet is almost completely protected by cathodic protection. Voltage is around 12 volts and current

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is 10 to about 20 amp for a bar-screen and 20 to about 30 amp for a rotary screen. An ordinary iron screen can be used as long as there is cathodic protection. A sea-water pump should be protected in the same way. Zinc anode is located on mouth-piece of pump. A sea-water pipe is also protected, but if the radius of the pipe is less than 5 in. the method cannot be applied.—MR. 17530

5.2.2, 6.2.2

On the Effect of the Corrosion Protection by Zinc Anode on the Strength of Corrosion Fatigue. (In Japanese.) K. Yamaguchi and H. Muramoto. J. Japan Soc. Testing Materials, **6,** No. 46, 474-478 (1957) July.

Galvanic anodes are used for preventing damages due to corrosion fatigue in hull or machine parts. An experiment was conducted to determine the effect of corrosion protection provided by this method on the corrosion fatigue of cast iron. Two kinds of cast iron in 3% saline solution were used. It was found that complete corrosion protection was brought about by keeping the anticorrosive potential of cast iron in 3% saline solution at about —0.95 V (S.C.P.); the corrosion fatigue could be reduced by this protection method to be about equal to that of corrosion fatigue in the air.—JSPS. 17980

5.2.3, 5.2.2, 8.9.3

What Trans-Canada is Doing to Protect Its System Against Corrosion. L. F. Heverly. Petroleum Engineer, **31,** No. 3, D22-D27 (1959) March.

The cathodic protection system in Trans-Canada Pipe Lines, Ltd.'s corrosion control program consists of rectifiers placed at 20-40 mi. intervals with magnesium anodes where power supply is unavailable. During construction, test leads were installed at 1 mi. intervals and at all crossings, and insulating flanges were installed at 80 mi. intervals, at compressor stations, etc. Spacing insulators, end support cradles, and casing end seals were used to prevent shorts between casing and carrier pipe. Interference from nearby structures was avoided by maintaining a 24 in. clearance and double coating. 17718

5.2.3, 8.9.5, 5.4.5, 3.6.6

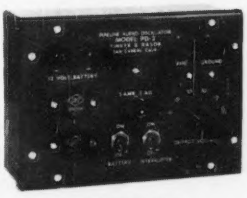
Hull Protection by Impressed Current. E. P. Anderson and T. P. May. Marine Eng. Log, **64,** No. 1, 39-41, 98, 100 (1959) Jan.

Detailed description of CAPAC cathodic protection system, developed by Charles Engelhard, Inc., which incorporates platinum-clad anodes and features automatic current-output control. System is designed to be used in conjunction with protective coating usually employed in hull protection. CAPAC system has sufficient latitude of control to accommodate increases in exposed steel surfaces as might occur with holidays and damage to paint film, and it will take care of any galvanic effects between dissimilar metals usually found in stern area. Active part of anode is disk of platinum alloyed with palladium. Various types of CAPAC equipment available, their application and economics involved are discussed.—INCO. 17624

5.2.3, 8.9.3

Cathodic Protection of Submarine Pipelines. (In Japanese.) Susumu Suzuki and Katsutomo Ohya. Corrosion





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Engineering, **7**, No. 4, 33-36 (1958) July.

Cathodic protection has been applied to two cases of submarine pipeline which connect between offshore tanker and tank in the refinery. One of them consists of a bare steel pipe and an asphaltic jute wrapped pipeline having 10-inch in diameter and 1,700 ft in length. Another consists of a 4-inch in diameter and 10,000 ft in length, a 8-inch in diameter and 4,500 ft in length, and two 10-inch in diameter and 10,000 ft in length bitumastic enamel coating pipelines.

Installations of cathodic protection on these pipelines were designed by considering the following points: 1. effect of the position of anodes on the current distribution (calculation and model test), 2. difficulties for setting of anodes, 3. difficulties for management of installations, 4. effect of the protective current on adjoining structures.

In the first case, one selenium oil-immersed rectifier rated at 16 volts 100 amperes dc and fifteen magnetite anodes (2½-in. in diameter, 33-in. in length) has been used to supply the protective current. This installation was completed in October, 1955, and full protection has been accomplished after 6 months.

In another case, one selenium oil-immersed rectifier rated at 10 volts 64 amperes dc and eight magnetite anodes (2½-in. in diameter, 33-in. in length) has been used. This installation was completed in January, 1957, and full protection has been accomplished after only 24 hours.

After measuring of the potential distribution we studied the relation of the current distribution with calculation, model test and actual data.

The initial cost of these installations of cathodic protection was 840 yen/sq. meter in the first, and 280 yen/sq. meter in another. 17740

5.3 Metallic Coatings

5.3.2

Protection of Steel by Zinc and Aluminum Spraying. (In German.) T. K. Ross, E. L. Smith and R. E. Mansford. Werkstoffe u. Korrosion, **9**, 755-760 (1958) Dec.

Properties of coatings obtained by the powder and wire methods. Good protection is achieved against atmosphere and corrosion under water when the metals are used on soft steel. Electrochemical behavior of base metal and coating. 17 references.—MR. 17580

5.3.2

Aluminized Steel, Properties and Uses. K. G. Coburn. Armco Steel Corp., Middletown, Ohio. J. Metals, **11**, 38-39 (1959) Jan.

Aluminized steel is manufactured in two grades. One has a silicon-aluminum alloy coating and is best suited in an environment where a combination of heat and corrosion is involved. The second grade has a pure aluminum coating and has excellent resistance to atmospheric corrosion. The physical properties, welding, corrosion and uses of these alloys are described.—NSA. 17680

5.3.2

Corrosion Prevention by Cadmium Plating. P. F. Norrish. Corrosion Prevention and Control, **4**, 51-53 (1957) August.

Protective action of cadmium coatings on steel, comparison of cadmium and zinc coatings; electroplating process and uses of cadmium-plated products.—MR. 14752

5.3.4, 6.4.2

Surface Treatment and Finishing of Light Metals. Pt. XII-E. Plating on Aluminum—Hard Chromium and Non-Electrolytic Deposits. S. Wernick and R. Pinner. Metal Finishing, **55**, 55 + 5 pp. (1957) May.

Chemical etching; chromium plating; tin immersion deposits; immersion silver deposit; chemical nickel plating.—BTR. 16384

5.3.4, 5.9.4, 8.8.3

Experiences in Electroplating and Related Processes. J. Chadwick. Electroplating and Metal Finishing, **11**, No. 7, 225-228 (1958) July.

Discussion of author's experiences in cadmium, zinc, nickel and chromium electrodeposition, electroplating of aluminum and anodizing of aluminum and its alloys.—INCO. 16790

5.3.4, 6.2.5

Harder Stainless Practical. R. N. Libsch. Am. Bosch Arma Corp. Steel, **142**, No. 26, 85-86 (1958) June 30.

Description of a method of nitriding which uses both a salt bath and gas treatment and which can be applied to all types of stainless steel. Photomicrographs.—INCO. 16542

5.3.4

The Application of Electrolytic Tinplate. W. R. Lewis. Werkstoffe u. Korrosion, **8**, No. 8/9, 456-462 (1957).

The introduction of continuous strip rolling and electrolytic tinning is described. Individual properties are given of the five layers: steel, $FeSn_2$, tin, oxide and oil. Causes of soldering defects are listed. The conditions which cause rusting of containers during storage and sterilization, and the corrosion of the insides of tins are reviewed. Protection of uncoated steel by electrical contact with tin pieces is illustrated as well as the life of various tin-coated steels in contact with plums. A final section deals with lacquering and the use of lacquered and unlacquered parts in one tin. A differential tinplate is described which has on one side 2.8 and on the other 11.2 g/m^2 tin for the outside and inside of the container. 9 references.—MA. 15825

5.3.4

Electrolytic Deposition of Lead-Nickel Alloy From Chloride-Fluoride Solutions. (In Russian.) K. M. Tiutina and N. T. Kudriavtsev. Doklady Akademii Nauk SSSR, **115**, No. 3, 580-582 (1957) July 21.

Method of depositing lead-nickel alloy containing up to 65 percent lead. Composition of the alloy. Its corrosion-resistant properties.—BTR. 15124

5.3.4

Corrosion and Corrosion Resistant Coatings. J. H. Payne. Inspection Engineer, **21**, 2-10 (1957) Jan.-Feb.

Examples of corrosion, methods of corrosion prevention, including metallizing.—MR. 16252

5.3.4, 6.3.17, 4.6.1

Metallic Coatings on Uranium by Sputtering and Evaporation. W. D. Davis. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn., KAPL-100(Del.), April 1, 1954 (Declassified with Deletions Feb. 25, 1957), 35 pp. Available from Office of Technical Services, Washington, D. C.

Adherent corrosion-resistant metallic coatings have been applied to uranium by a combination of cathodic sputtering for cleaning the uranium surface and evaporation or sputtering to deposit the coating metal. Evaporated chromium coatings gave the most satisfactory results and, with suitable heat treatment, resisted corrosion in boiling water for an average of over 200 hours. Sputtered coatings were inferior to evaporated coatings. (auth).—NSA. 15752

5.3.4, 6.2.3

Galvanized Structural Steel in Gulf Coast Construction. J. W. Sicard. Ind. Eng. Chem., **49**, No. 12, 69A-70A (1957) Dec.

The Celanese Corporation of America has used galvanized structural steel for their new polythene plant on the Gulf Coast. Estimates showed that galvanizing would cost either the same as or less than painting, depending on the preparation used. All but 5 per cent of the structural work was designed to be single-dipped in 30 ft baths, and was assembled with galvanized bolts and nuts wherever possible, instead of by welding. 1 table.—ZDA. 16290

5.3.4

Vacuum Metallising. D. J. T. Kennedy. Ind. Finishing, **9**, No. 107, 554, 556-559 (1957) May.

The principles of the process of vacuum metallizing, plant maintenance and equipment are described. The process consists basically of three main steps: coating the workpiece with lacquer and baking this to give a hard glossy undercoat, evaporation and deposition of metallized coating and coating with lacquer to protect the newly deposited film. The undercoating is usually of baked alkyd resin. The top coat lacquer may be dye tinted, though the normal requirement is a plain water-white transparent lacquer.—RPI. 14675

5.3.4, 1.2.2

The Electroplating of Screws and Other Small Articles. Pt. IV. Quality Control and Costing. (In German.) W. Peters. Draht, **8**, No. 4, 127-131 (1957) April.

Inspection for plating thickness, corrosion resistance and, where necessary, for the dimensions of screw threads is discussed. The stripping of defective coatings and effluent treatment are described. Costs and their estimation are dealt with and an example is worked through in detail. 2 tables.—ZDA. 16233

5.3.4, 8.1.2

Galvanized Steel in Building and Public Works. (In French.) J. R. Rouff. Corrosion et Anticorrosion, **6**, No. 6, 211-216 (1958).

Characteristics, performances and protective mechanisms of zinc coatings on iron and steel are considered and present and potential uses in buildings are reviewed.—MA. 17578

5.3.4

Effect of Electroplating Conditions on Structure and Wear Resistance of Iron Deposits. (In Russian.) Yu. N. Petrov. Metalloyed. I. Obrabotka Metalloy., No. 12, 53-56 (1958) Dec. Translation Available from: Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, Calif.

Study of most favorable conditions of electrolysis for dense, fine-grained and wear-resisting iron deposits, able to meet fully all repair requirements. Com-



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position of baths used. Effect of limited amounts of organic bath additives on density and wear resistance of plates. Current density and electrolyte temperature as affecting the results. Plating solutions recommended for repair purposes, based on subsequent performance of repaired tractors in service. 3 figures. —HB. 17549

5.3.4, 2.3.2, 2.2.5

Copper-Nickel-Chromium and Corrosion Protection. A. H. DuRose and W. J. Pierce. Harshaw Chemical Co. Metal Finishing, **57**, No. 3, 44-50, 54 (1959) March.

Presents some general conclusions, based on results of numerous tests—accelerated, roof and service—involving thousands of plated panels over the years. Discounting extreme recesses and taking into consideration supposedly more corrosive atmosphere, plated parts (including automotive decoration) are as good as they were in 1938. Between different types of outdoor exposure sites order of merit of different copper-nickel-chromium coatings is usually the same, with some occasional unexplainable reversals. Similar differences in order of merit are obtained between outdoor exposure sites and accelerated corrosion tests as are obtained between different outdoor exposure sites. Copper undercoat prior to grey nickel or sulfur-free nickel deposit is of practically no benefit. Copper undercoat prior to sulfur-containing nickel is frequently beneficial, improvement being equal to that of equal thickness of additional sulfur-containing nickel. When corrosion does occur, copper-nickel-chromium plated article will be more unsightly due to crater-rusting than would the case for nickel-chromium. Sulfur-containing semibright or bright nickel deposits do not exhibit as good corrosion protection as do sulfur-free deposits. Conclusion on chromium is that distinct advantage in corrosion protection can be obtained by using high temperature chromium solution or crack-free variety, but heavier than normal thickness should be used. Evaluation of corrosion results, methods of rating (ASTM #, General Motors Performance Index) and factors leading to variability of results, are considered. Results on nickel deposits and on chromium deposits and measurement of chromium thickness are discussed. Tables summarize results for various sites (Florida, Detroit, Pittsburgh, Kure Beach, New York and Cleveland); and for Corrodkote, acetic acid salt spray and accelerated acetic acid salt spray tests. Photomicrographs show surface pitting and pinholes.—INCO. 17844

5.3.4, 3.2.2

Pores and Pitting of Plated Deposits. J. M. Odkerhen. Metal Finishing, **56**, 64-66 (1958) Oct.

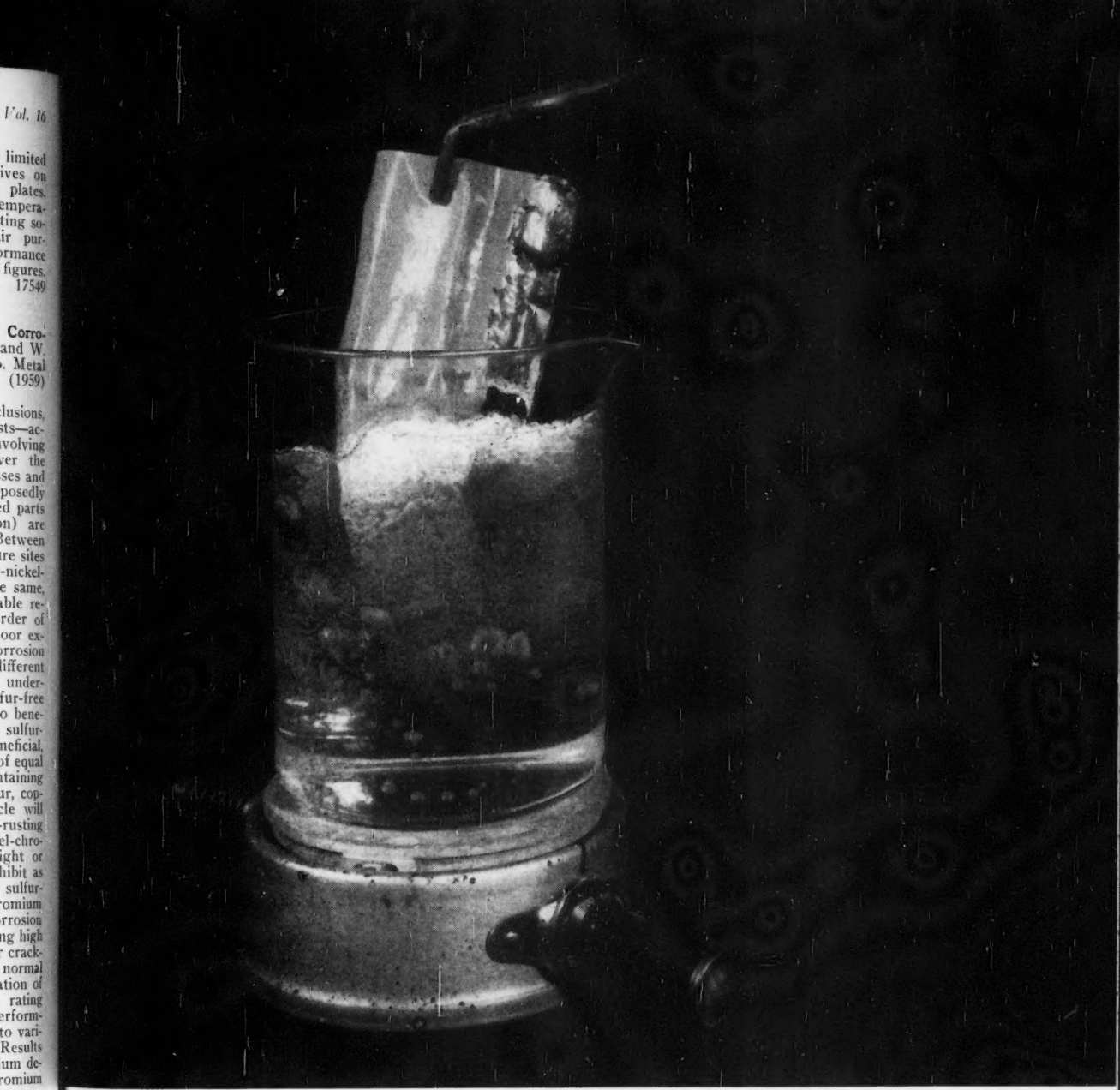
Pores and pitting can be attributed to base material—porous base or poor mechanical pretreatment or occlusions in the material; chemical pretreatment; plating solution—suspended material or dissolved impurities.—MR. 17512

5.4 Non-Metallic Coatings and Paints

5.4.3, 6.6.7, 6.6.8

How to Protect Metals with Elastomeric Linings. L. A. Woener. Materials in Design Eng., **46** No. 2, 94-98 (1957).

Principal elastomeric lining materials are described—natural rubber and syn-



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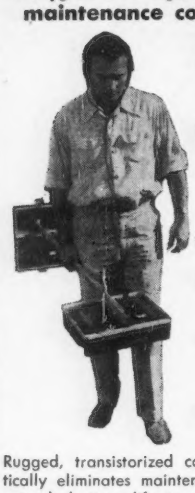
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thetics which include Neoprene, nitrile and butyl rubbers, and Hypalon. Although not an elastomer, polyvinyl chloride is included. Their resistance to a wide range of chemicals at normal and elevated temperatures is discussed and tabulated.—MA 17938

5.4.5, 6.2.3

Paints for Structural Steel. W. J. Bradley. American Paint Journal, **43,** No. 18, 70+4pp. (1959).

The type of shopcoat primer must be considered, with the type and degree of surface preparation. No single type of painting system can be recommended as being outstanding in meeting all the specific properties and requirements.—RPI. 17750

5.4.5, 3.4.8

Investigation of Anticorrosive Properties of Surface Coatings for Equipment Used to Remove Hydrgen Sulphide from Gas by Means of Sodium Arsenate. (In Russian.) G. A. Isakov and Y. D. Kernos. Gazovaya Prom., No. 7, 21-23 (1957); Chem. Absts., **52.** No. 5, 4208d (1958) March 10.

A number of coatings are discussed, such as laquers and risens, which are referred to only by their Soviet trade names. The best material found for coating the inside iron scrubbers was a resin with a ferricoxide filler.—RPI. 17952

5.4.5

Solvent-Free Epoxy/Amine Combinations in Surface Protection. (In German.) H. Zumstein. Fette, Seifen, Anstrich, **60,** No. 7, 547-552 (1858).

The film properties of epoxyresin/polyamide resin combinations cured with polyamines, usually in the cold, but also at high temperatures, are reported. The amines are characterized by possession of tertiary amino, and at least one phenolic, groups, Alternatively, triethylene-tetramine was used. Epoxy resins of viscosity 100 poises in combination with polyamide resins provide soft films, other film properties being substantially unchanged; addition of solvent to an epoxy resin so employed to reduce its initially higher viscosity to the same value, results in cured films which are hard but susceptible to solvent attack. Fields of application, e.g., in primers and protective coatings generally, with typical formulations are given.—RPI. 17673

5.4.2, 5.4.7, 3.5.9

Application of New Metal Coating Which Withstands 2000 F. J. F. Rice. Ind. Heating, **26,** No. 3, 554, 556, 558, 560-561 (1959) March.

New resin-ceramic protective coating for metal is easily applied, is light, durable, hard and yet flexible, and withstands temperatures up to 2000 F for extended periods. Designated PT-404, coating was developed for missile use but has many other potential uses. Coating has withstood extensive testing including application to marine engine mufflers. Coating is applied by brush or spray gun and then baked.—INCO. 17811

5.4.2, 3.5.9, 3.2.2, 6.2.1

High-Temperature Corrosion Protection by Ceramic Coatings. (In German.) Armin Petzold. Werkstoffe u. Korrosion, **9,** 761-765 (1958) Dec.

Protection of carbon steel up to 700 C and alloy steel up to 1000 C against oxidation and intercrystalline corrosion by high-temperature enamels. Heat resistant aluminum oxide, zirconium dioxide, titanium dioxide, mullite, porcelain or nickel-Magnesium oxide-cermet coatings.— RML 17526

5.4.5

Protective Organic Coatings for Steel. Pt. II. Louis J. Nowacki and E. R. Mueller. Industrial Heating, **25,** 2308+4 pp. (1958) Nov.

Describes finishing systems for coating steel, including the proper preparation of the steel surface for painting; the right combination of coating materials including primers, primer-surfacers, fillers, and top coats; the correct application of the coating materials and proper curing of the coatings.—BTR. 17518

5.4.5, 2.2.2

Weathering Properties of White Lead and Zinc Oxide Paints. J. R. Rischbieth and K. R. Bussell. J. Oil Col. Chem. Assoc., **41,** No. 12, 862-868 (1958) Dec.

Authors describe investigations into the effect of white lead and chalk-resistant zinc oxide, used singly and mixed together in the proportions 2:1 and 1:2, on the durability of linseed oil paints. Three-coat systems were exposed on wooden and steel panels for 4 years at Cape Schanck, 5 years at Maribyrnong and 5½ years at Villawood in Australia. It was found that zinc oxide as the sole pigment was satisfactory in a finishing paint but was inferior in a priming paint, whereas the reverse was true for white lead. The best results were obtained by using mixed pigments in each coat, but with white lead predominating in the primer and undercoat and zinc oxide predominating in the finishing coat. 4 tables, 2 figures.—ZDA. 17581

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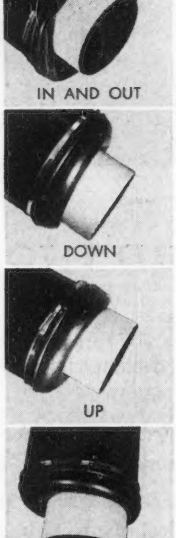
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5.4.5

New Developments in Wash Primers. M. H. Roth and M. D. Kellert. Official Digest, Federation of Paint and Varnish Production Clubs, **30**, No. 404, Pt. I, 980-988 (1958).

Modification of a standard wash primer formulation by addition of other resins has shown that for early development of water resistance, good adhesion and freedom from blistering in multicoat systems (alkyd finishing coats, generally), a phenolic resin was most useful.—RPI 18018

5.4.5

A Review of Organic Coatings. Harold A. Pollay. Products Finishing, **23**, No. 2, 50-55 (1958) Nov.

A description of organic coatings commonly used by industry today, together with a discussion of the properties of each.—BTR. 17564

5.4.5, 8.9.5

Developments in Ships' Protective Coatings. F. Perkins. Corrosion Technology, **5**, No. 8, 247-249 (1958) August.

Analysis of sources and mechanisms of corrosion with methods of preventing the electrochemical reaction. Polymers, vinyls, bituminous materials and chlorinated rubber for underwater coatings; top-side paints; primers and paints that give diversity of service and protection.—MR. 17534

5.8 Inhibitors and Passivators

5.8.1, 5.6.1

Interactions Between Four Volatile Corrosion Inhibitors. R. L. LeMar. Rock Island Arsenal, U. S. Army, December 1957, 42 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 151010).

Volatile corrosion inhibitor papers should not be mixed under pressure in the packing of military material, according to this report. The mutual compatibility of four VCI-on-Kraft papers were evaluated in pressure contact and vapor mixtures to compare the corrosion inhibition afforded steel panels by the single papers and their combination. Results indicated inadequate compatibility between papers under pressure contacts, that a binder such as casein contributes to staining, and that high humidity increases incompatibility in contact situations. The results of vapor phase mixing tests, however, indicate complete compatibility of the vapors of the four VCI papers, thus allowing mixing where vapors are utilized.—OTS. 17922

5.8.2, 6.2.2, 4.6.5, 4.6.6

Calcium Cyanamide as an Inhibitor of Iron Corrosion in Aqueous Solutions. (In Polish.) Stefan Pawlikowshi, Iwo Pollo and Jerzy Wegiel. Zeszyty Naukowe Politechniki Slaskiej, No. 12, 107-128 (1957).

Influence of calcium cyanamide on the corrosion rate and change of the irreversible electrode potential of iron (0.3% carbon) in distilled and tap water and in series of salt solutions in relation to underground corrosion. Addition of 4% calcium cyanamide to tap and distilled water reduced corrosion rate ten times. Greatest inhibition was observed in calcium sulfate solution. Inhibition of corrosion rate in water and the majority of salt solutions by calcium cyanamide additions is caused by the hydrolysis of the latter and by the rise in alkalinity of the solutions.—RML. 17895

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5.8.2, 4.6.2, 6.2.5, 3.2.2, 5.7.7

Investigation of Chemical Inhibitors for Stress-Corrosion Cracking of Stainless Steel. Summary Rept. for Jan., 1954 to June 1957. F. E. Clarke and A. J. Ristaino. September 9, 1957, 18 pp. Naval Engineering Experiment Station, Annapolis.

Since recommending coordinated phosphate-pH control for the Submarine Thermal Reactor boiler in 1950, numerous laboratory tests, experimental boiler tests, and actual service tests have been made of this treatment with and without supplementary chemical inhibitors, to determine its merit in maintaining proper waterside conditions and particularly in preventing stress-corrosion cracking. It has been determined that the treatment originally recommended affords adequate protection for both mild steel and stainless steel surfaces completely submerged in boiler water. However, stainless steel parts exposed intermittently to the boiler water and its vapor are not protected against stress-corrosion cracking if both dissolved oxygen (or certain oxidizing agents) and chloride ion are present. There is evidence that caustic alone will cause cracking under the same conditions. This damage has been prevented by supplementary treatment with sodium sulfite oxygen scavenger. A more practicable inhibitor for the stress-corrosion problem still is being sought. (auth)—NSA. 17865

5.8.3, 6.2.2, 4.3.2

Some Observations on the Mechanism of Inhibition. A. C. Makrides. J. Electrochem. Soc., **106**, No. 1, 7-9 (1959) January.

Thiocyanic and citric acids are efficient inhibitors of dissolution of iron in dilute nitric acid solutions. It is concluded that inhibition does not generally involve 'poisioning' the combination step in the hydrogen evolution reaction. Some observations on the kinetics of dissolution of iron in dilute nitric acid solutions are also presented.—EL. 17802

5.8.3, 3.8.3

Electrochemical Study of Inhibitors. Pt. 1. Neutral Solutions of Sodium Chloride. (In Italian.) Dina Gherard. Metallurgia Italiana, **51**, 13-20 (1959) January.

Theory of electrochemical action of inhibitors; interpretation of polarization curves. Corrosion potentials, polarization data and weight losses obtained from tests on steel (0.09% carbon, 0.02% sulfur, 0.102% phosphorus, 0.28% manganese) with series of organic and inorganic inhibitors. Potassium dichromate and potassium nitrite were most effective of inorganic inhibitors; of others, only Teramin showed any clear inhibitor action, chiefly due to anodic polarization.—RML. 17716

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Cathodic Protection in An Urban Area With a Distributed Anode System*

By JAMES P. McARDLE, JR.

Abstract

Discusses problems involved in applying cathodic protection to cables in an underground conduit system in an urban area where resistivity conditions are not uniform and where other subsurface structures are present. Distributed anode system was used in tests described. Impressed current was supplied by three small rectifiers. About 21 sheath miles of cable were protected of which 16 miles were bare lead sheath. 5.2.3

Introduction

PROBLEMS INVOLVED in designing cathodic protection in open territory usually are not too difficult. In urban areas the difficulties are increased by local conditions such as lack of suitable space for a ground bed and presence of networks of other subsurface structures, particularly when these structures are not coated and are in the vicinity of the ground bed and the cable. Because avoidance of damage to other structures must be one objective, design of protection in urban areas often is a difficult matter.

This article discusses problems involved in applying cathodic protection to underground conduit cables in an urban area where resistivity conditions are not uniform and where other subsurface structures are present.

Concentration of earth current should be avoided, so far as practicable, for three reasons: (1) current which increases negative potential of a lead cable to a value beyond that required for adequate protection is wasted, (2) lead sheath may be subject to corrosion from alkaline salts under certain conditions involving high negative potentials and (3) the smaller the negative potentials, the lesser the chance that potentials of other structures will be affected.

To minimize current requirements, the cable sheath must be isolated from other metallic structures except where drainage bonds are required to protect these structures. It is desirable that the sheath be no more negative than required to insure protection. The method described in this article is applicable to cables in non-metallic duct but is not intended to protect cables in iron ducts.

Contacts between structures limit the effectiveness of cathodic protection on a specific structure. Bonding may be required, however, if current from the protection system is impressed on the other structures unless they are to form a part of a coordinated protection system.

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About the Author



JAMES P. McARDLE, JR., is engaged in corrosion work with American Telephone and Telegraph Company, where he has worked since graduation from Alabama Polytechnic Institute in 1926 with a BS in electrical engineering.

Therefore, anodes discharging current should be placed so as to minimize the chance that significant amounts of current will be picked up by unprotected structures. The smaller the current picked up, the less the requirement of drainage from these structures.

The calculated resistance to the flow of current through a grounded electrode into the surrounding earth is shown in Figure 1. The greater part of the resistance is close to the electrode. If the resistance is plotted against distance of measurement from the electrode, the resistance rises rapidly at first and then becomes less rapid until it finally becomes constant.

If the positive terminal of a battery is connected to a metallic rod or electrode and the negative terminal to a grounded structure 500 feet or more away, the potential gradient in the earth immediately surrounding the electrode (anode) will have a very steep slope. Near the center the gradient is steep; the slope becomes more gradual with distance until at some distance away the terrain becomes flat.

The current return, or negative, electrode (cathode) similarly is surrounded by a potential field; but because current flow through the earth is toward, rather than from, this electrode—the surrounding earth gradient potential slopes in the opposite direction. Thus the positive electrode may be represented as a peak and the negative electrode as an elongated depression. If the two electrodes are far enough apart, a level plain will lie between them and they will be remote from one another.

In the usual arrangement of cathodic protection, the negative side of a source of d-c potential is connected to the structure to be protected and the positive side to the anode. Current enters the earth at the anode and then fans out, distributing itself along the protected structure. As the distributed current is picked up by the structure, voltage drops produce two effects: (1) potential gradient in the earth surrounding the anode produces an anode effect and (2) potential gradient in the earth surrounding the structure under protection is changed, producing a structure effect. Thus, drainage action can be regarded as two separate actions.

If the anode is remote from its electrical connection to the structure, the effect of the anode in changing the potential gradient in the earth in the vicinity of the structure is small and the only practical effect is on the structure to earth potential. If the anode is close to its connection to the structure or to some other point on the structure, the anode effect in changing the earth potential gradient around the structure can be considerable.

If the structure to be placed under protection is in the area of the peak created by the anode, the potential gradient will be distorted by presence of the structure. Likewise, the presence of other metallic structures will distort the gradi-

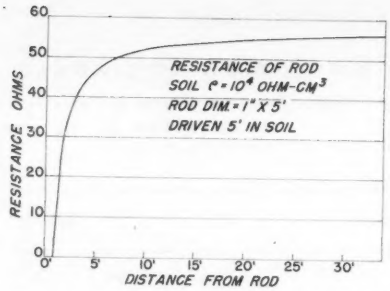


Figure 1—Calculated resistance to current flow through a grounded electrode into surrounding earth.

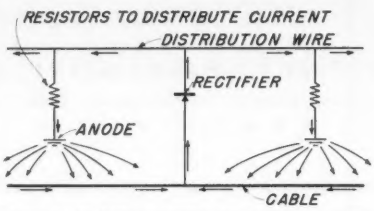


Figure 2—Cathodic protection by distributed anodes.

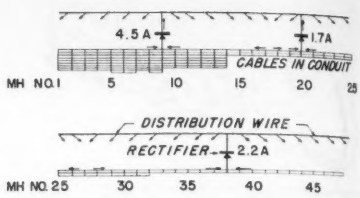


Figure 3—Layout of the protection system.

ent, because their conductivity is greater than that of the earth.

Where earth conditions are not uniform, e.g., a low resistivity stratum overlies a high resistivity stratum, the distance the anode must be placed from the protected structure and from other metallic structures to minimize effects resulting from anode proximity can be several thousand feet. Such conditions exist in Greensboro, North Carolina.

In the Greensboro area, earth resistivity increases with depth. At the upper levels (5 to 30 feet) it varies widely from point to point in a range of about 3000 to 50,000 ohm-centimeters. At levels below about 50 feet, it increases with depth, from 500,000 to 800,000 ohm-centimeters at rod spacings of 3000 feet.

Because such high resistivity is encountered at the lower levels, currents discharged to ground from a point-type or concentrated anode tend to spread out over a rather wide area. Hence, these currents are picked up by neighboring underground structures lying in their path.

Tests have indicated that the potential field resulting from a single point-type anode placed at any point in the Greensboro urban area and discharging a relatively large current sufficient to provide satisfactory cable potentials will be extensive and will have appreciable effect on foreign subsurface structures situated between the ground bed and the cable, even if the ground bed is located as far away as one mile.

On more than one occasion when a trial anode for cathodic protection of underground cables was placed within a few hundred feet of other subsurface structures, tests indicated that most of the ground current was intercepted by these subsurface structures. The subsurface structure network thus served to augment the ground bed, affording excellent protection to the cable but with obvious detriment to the other structures unless they were included in the protection system.

These tests showed that any impressed current ground bed concentrated at one point and delivering current sufficient to provide adequate protection to the many cables in the duct system should not be installed in the urban area. It would be necessary that such a ground sytem be located several miles from the city. This would be impracticable in this case.

If, however, the anode were placed within a few feet of the cable, the cable would be in the potential field area of the anode and would, therefore, pick up current due to anode proximity. Other subsurface structures, if they were located at or near the edge of the anode potential gradient, should pick up only a small portion of the current. Potential gradient effect of such an anode on other structures should be small if the current and the distance between the anode and cable are made small.

One method of accomplishing this would be to install anodes in the duct line. Such an arrangement would not have been economical for the situation at Greensboro because of the length involved and might not have afforded adequate distribution of current because of possible variations in the resistance of individual anodes in the duct line.

Tests indicated that application of sufficient protective current could be accomplished economically by ground electrodes placed within a few feet of the conduit system at about 130-foot intervals.

Under favorable soil conditions, magnesium anodes would be ideal for this purpose. In this locality, however resistivity conditions would restrict output of the individual magnesium anodes to a value insufficient to insure protection. Therefore, a distributed ground system was used with an impressed current.

Because only small currents were to be used, consideration was given to the use of iron rods or pipes for anodes. However, dissipation rate of this metal would limit anode life to a relatively few years. For example, a 1-inch by 5-foot rod or a 1½-inch by 5-foot pipe would be expected to last not more than five years. Replacement costs could be considerable, particularly if the surface area above the anode were subsequently paved over. Consequently, high silicon cast iron rods were considered preferable because of the longer life expectancy of these rods.

Cables in the area under consideration are in tile conduit. With this type construction, they are in direct contact with the surrounding soil only by silt and water penetrating through the duct joints. A pipe network in the area is in contact with the soil and likely to pick up an appreciable portion of the current from an impressed current ground system unless the anodes were located at a point remote from the cable and the pipes, i.e., unless the pipes were outside the potential gradient created by the anode. Spacing of distributed anodes was governed by current density requirements, availability of pole line facilities on which to place a distribution conductor and possible interference to other structures.

By suitable arrangement of the distributed anodes, effect of the current from each anode is confined to a small area, particularly when the anodes are placed close to the cable being protected and small currents are utilized. Tests indicated that it would be possible to install the anodes at Greensboro in relatively close proximity to foreign metallic structures without adversely affecting such structures. If the anode is a rod buried vertically in the earth to a depth of 5 feet, about 90 percent of the potential gradient around the anode was found to be distributed in an area within 10 feet of the anode, under average conditions.

Use of this type anode system resulted in a more uniform effect on the structure being protected; consequently, high current density near the drainage point could be avoided. Tests indicated that the system would require in most cases

TABLE 1—Anode Current Distribution

Manhole No.	Section Length	No. Cables		No. Anodes	Avg. Amp. Per Anode
		Lead	Polyethylene		
1– 5	1096′	9	5	8	.118
5– 7	373′	10	4	5	.131
7– 8	426′	9	5	4	.099
8– 9	419′	10	4	1	.173
9–14	2401′	8	5	13	.092
14–18	1908′	3	3	12	.078
18–32	6748′	3	..	48	.049
32–48	7203′	2	..	49	.036
	Total 20,574′			Total 140	Total .060

TABLE 2—Sheath Current Distribution

Manhole No.	No. Bare Lead Cable	Average Current (Amp)	
		100 Ft	100 Sheath-Ft
1– 5	9	.086	.0095
5– 7	10	.175	.0175
7– 8	9	.093	.0103
8– 9	10	.041	.0041
9–14	8	.050	.0062
14–18	3	.049	.0164
18–32	3	.035	.0116
32–48	2	.024	.0122
	Totals .041		.0103

less than 100 milliamperes per anode for satisfactory improvement in cable-to-soil potentials.

At Greensboro, distributed anodes could not be placed economically throughout the area to be protected. A 1800-foot section adjacent to the central office is in a downtown business area where local conditions and restrictions precluded placing the anodes outside the duct system. Beyond this area, however, conditions were favorable; the conduit generally is paralleled within a few feet by a pole line located in a grass plot between the sidewalk and curb throughout the proposed system's length. In two sections (1900 and 3700 feet) the pole line is on the side of the street opposite the conduit system, with water and gas pipes under the street surface between the anodes and the cable.

Figure 2 shows the method of distributing current to the anodes. The anodes, high silicon cast iron, 1 inch in diameter and 5 feet in length, were placed vertically in holes augured adjacent to selected poles, one rod to each pole. Because current requirement per rod would be small, only soil backfill was used. A total of 155 anodes was installed, interconnected by a continuous length of two No. 14 AWG insulated wires placed aerially on the pole line between Manholes 1 and 48, a distance of 20,500 feet. One No. 14 insulated connecting wire was placed down the pole between the aerial conductor and each anode, and a Holloway shunt inserted in this wire to enable the current to be measured. The down wire was covered with wood molding to a height of 6 feet above ground.

Considerable variation was found in ground resistance between individual anodes. Where the current to an anode was considerably greater than that to adjacent anodes, an auxiliary resistance was placed in the connection to the anode. This resistance, a wire-wound spool type, was inserted in the down wire on the pole between two bridging connectors, the down wire and anode lead having been initially connected by a bridging connector to permit measurement of individual anode resistance pending final adjustment of the system.

Minor adjustments may be required after the system has been in operation for a short period. After final adjustments have been made, leads from the resistance will be soldered to the down wire and the anode lead, and the bridging connector above the molding replaced by a sleeved connection.

Over-all resistance of the cables to anodes in parallel was about two or three ohms when measured at several points along the system. The resistance increased to about four ohms after auxiliary resistances were added in the down wires to the anodes to equalize the anode currents. Because over-all resistance was low, voltage requirements also were low and current could be supplied by small rectifiers.

General layout of the protection system is shown in Figure 3. Three selenium rectifiers, spaced to provide optimum coverage, were mounted about four feet above ground on poles adjacent to manholes at selected locations. Positive terminal of each rectifier was connected to the anode distribution wire and the negative to the cable sheath. The rectifier at Manhole 9 was rated at 18 volts and 6 amperes; the rectifiers at Manholes 20 and 38 were rated at 18 volts and 3 amperes.

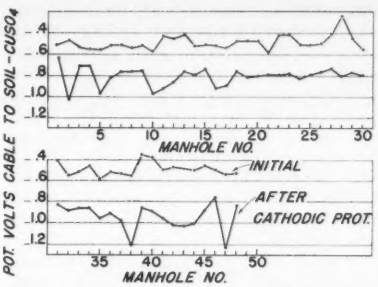


Figure 4—Cable-to-soil potentials at manholes before and after application of cathodic protection.

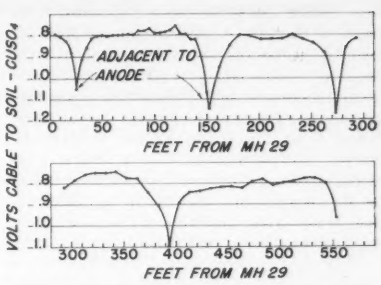


Figure 5—Cable-to-soil potentials in area between two adjacent manholes.

TABLE 3—Rectifier Current Distribution

	RECTIFIER			
	MH 9	MH 20	MH 38	All
Output Current Amp.	4.47	1.65	2.25	8.37
Manholes Covered	1-17	17-27	27-48	1-48
Distance Kilofeet	6.2	4.6	9.8	20.6
Sheath Feet Covered				
Bare Lead—Kilofeet	42.2	13.9	22.1	81.2
All Cables—Kilofeet	72.4	15.2	22.1	109.7
Average Current Amp. per 100 Ft				
Bare Lead Cables	.0106	.0119	.0102	.0103

In Table 1 the number of anodes and the average current per anode are shown by divisions based on the number of cables present. Sections with the larger number of cables have higher anode currents. Average current per anode of the 140 anodes connected is 60 milliamperes.

Table 2 shows the distribution of sheath current. The average current per 100 sheath-feet is ten milliamperes.

Distribution of current by rectifiers is shown in Table 3. Because the distribution wire was continuous throughout the system, the effects of the rectifiers overlap. It will be noted that 109.7 kilofeet, or approximately 21 sheath-miles of cable, are protected by a current of 8.4 amperes. Combined wattage of the three rectifiers is approximately 200.

Figure 4 shows the cable-to-soil potentials at manholes before and after application of cathodic protection. The change in potential generally was .2 volt or more. Maximum changes occurred where a manhole was within about 10 feet of an anode and minimum changes where it was midway between two adjacent anodes. It was desired, so far as practicable, to obtain potentials at least 0.7 volt negative to insure protection against anodic corrosion and not more than 1.25 volt negative to minimize possibility of corrosion due to alkaline attack.

Figure 5 shows cable-to-soil potentials in an area between two adjacent manholes. In the section where measurements were made, there is a grass plot above the conduit system and the anodes are located about three feet from the cables, horizontally. Readings were made between the cable sheath and a copper sulphate electrode moved over the ground surface above the cables. Maximum negative potentials occurred when the electrode passed near an anode. The potential was not less negative than 0.7 volt at any point. In this section there were three cables in the conduit system, and the anode currents were 70 milliamperes or less.

In most cases, it was not possible, due to local conditions, to measure the drop in potential on the water or gas pipes; therefore, the amount of current picked up by these structures could not be determined precisely. In the section between Manholes 20 and 29, in which the water main is parallel to the conduit run between the anodes and cable, potential drop measurements could be made on the service pipes from one side of the street to the other at several locations where there were taps leading to opposite sides of the water main.

Tests were made by measuring the IR drop over a known length of the pipe with known values of current applied to the pipe and by measuring the change in voltage drop with the anode current interrupted. Calculations indicated that currents of one to seven milliamperes were picked up from the anodes by lateral pipes and carried in the direction of the cable. Although the amount of current could not be determined precisely, it generally was less than that normally present on the pipes attributable to natural causes.

At several points, an anode was originally located at the base of a pole at which the multigrounded power neutral wire is grounded. Although the anode and power neutral ground were only a

few inches apart at these locations, tests showed that little current was picked up by the neutral. One such point was located about 100 feet from one of the rectifier connections to the cables. For test purposes, the power neutral circuit was bonded to the cable at the drainage point. Under this condition, seven percent of the current delivered to ground at this anode was picked up by the neutral wire. After removal of the bond, the pickup was reduced to only two percent.

Pipe-to-soil potential readings were made at water meters and fire hydrants, the earth contact electrode being a copper sulphate half cell placed on the surface of the ground about one inch away from the structure. Readings were made with the rectifier current in the on and off position. Change in potential was a function of the amount of anode current and the distance between the pipe and the anode. In general, little or no change was noted where the pipe was 15 feet or more from the nearest anode. Where the separation was 10 feet or less, it was found desirable to reduce the current delivered to the anode or to disconnect the anode from the ground system.

It was not practicable to make sufficient tests on the gas pipes because these pipes were not generally accessible. Effect of the anode currents on the lateral pipes could be determined only by excavating to each pipe or by making a connection to the pipe at the meter inside the building served. Consequently, the influence on the gas system could not be readily determined. There was a possibility that, unless suitable drainage were provided, the pipes might be adversely affected at some points.

A solid bond was made to the gas main from the cable at Manhole 15. This manhole is centrally located with respect to the gas line exposure.

At this location the current drained from the gas pipes was 12 percent of the total output of the three rectifiers. It comprised 11 percent of the output of the rectifier at Manhole 9, 25 percent of the output at Manhole 20 and 6 percent of the output at Manhole 38. This distribution was approximately that which would be expected, based on the relative degree of exposure and the rectifier outputs in the various areas.

Tests made on the cable after installation of the bond indicated that adequate protection could be achieved without reducing the current drained from the pipe. Because of this, the small amount of power involved and the difficulties involved in determining how much reduction could be tolerated on the gas pipe, no attempt was made to reduce the drainage current.

If point-type, instead of distributed, anodes had been employed, the bulk of the ground current would have been intercepted by the water and gas pipes, requiring joint drainage connections and bonds between structures. Then, because the resistance to earth of the pipes was considerably lower than that of the cables, a higher percentage of the rectifier current would be drained from the pipes than from the cables. The arrangement would be uneconomical therefore, for cable protection unless the companies owning the other structures were willing to bear their proportionate expense of the costs of operating such a system.

Under the present arrangement, the current picked up by foreign structures is very small and can be drained satisfactorily by means of bonds with little increase in electrical power requirements for equalization of potentials on the foreign structures.

Any discussion of this article not published above will appear in the June, 1960 issue.

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Part 3—Underwater Corrosion of Ten Structural Steels

Corrosion of Metals in Tropical Environments*

By B. W. FORGESON, C. R. SOUTHWELL and A. L. ALEXANDER

Abstract

Corrosion of ten structural steels exposed to tropical sea and fresh waters has been evaluated following an eight-year exposure period. The severity of corrosion is compared between the natural tropical environments of sea water mean tide, and sea water and fresh water continuous immersion, and correlated with similar corrosion tests that have been made on the east and west coasts of the United States. Corrosion resistance for mild carbon steel is compared when exposed with millscale, pickled, and machined surfaces. Underwater corrosion rates are compared for unalloyed carbon steel, copper-bearing steel, steels containing small percentages of nickel and chromium, and proprietary low-alloy steels. A comprehensive evaluation of the measured and observed effects of corrosion is given for the ten steels following exposure in each of the tropical environments. 2.2.7

Introduction

AN EXTENSIVE investigation was initiated in 1946 to study the corrosion rates and characteristics of fifty different metals and alloys exposed to five natural tropical environments in the Panama Canal Zone. These environments included a marine and an inland atmospheric exposure, fresh water immersion, and continuous and mean tide sea water immersion.

Earlier short-term studies[1] indicated that it was necessary to make periodic measurements over a longer period of time in order to predict with accuracy the useful life, in the tropics, of the metals and alloys included in this study. Ten replicates of each metal were exposed in the five environments in order that duplicate panels could be removed from each location after exposure periods of one, two, four, eight, and sixteen years. The eight-year specimens were removed in the summer of 1955, thus providing the fourth point for corrosion-time curves.

An earlier report[2] incorporating the first of the eight-year data described in detail the methods and procedures employed in the program and devoted special attention to the corrosion rates of five unalloyed metals and a structural steel in the five natural tropical environments. A subsequent report[3] described the corrosion rates of ten different structural steels after eight years' exposure to tropical marine and tropical inland atmospheres. The present report is concerned with the corrosion of these same ten structural steels during continuous immersion in the fresh water of Gatun Lake, continuous immersion in the Pacific Ocean at a point more than a mile from the mainland on the Fort Amador Causeway, and exposure at mean tide elevation in the Pacific Ocean at the same Fort Amador location. The results of the study reported herein continue to widen the scope of knowledge of the corrosion rates of structural steels in natural environments.

Test Conditions

Exposure Environments

Chemical analyses of the water at each of the exposure sites are presented in Table 1. These data are summaries of individual analyses of samples obtained at the sites each March (during the dry season), May (beginning of the wet season), and November (end of the wet season) during the period May 1946 to March 1951. Fresh water and sea water were sampled at the elevations of the exposure racks.

The sea water at Fort Amador is similar in composition to sea water in other parts of the world as, normally, unpolluted sea waters vary only slightly in dissolved constituents between remote locations.[4] Fresh water, however, varies widely between one location and another —even in the same stream or body of water. Therefore such factors as oxygen concentration, pH, temperature, velocity, and nature and amount of dissolved solids may drastically affect the type and extent of corrosion. Thus the analysis of the fresh water of Gatun Lake must be considered carefully when correlating results obtained here with those of other investigators.

The average range of tide at the Fort Amador site is approximately 13 ft with a maximum tidal range near 21 ft. During the spring tide the velocity of water past the exposed specimens does not exceed 0.5 ft/sec. Even this maximum velocity is considered insignificant in its relationship to corrosion rates in these tests.[5] There is no appreciable flow of water at the Gatun Lake site. In the tropics the average annual temperature of the water is appreciably higher than in similar waters in temperate climates, and this temperature difference is significant in the interpretation of the results from these tests. Figure 1 presents water temperatures at each of the exposure sites.

Metals and Methods

The chemical compositions of the ten structural steels are presented in Table 2. Four of these steels were proprietary high-strength low-alloy types; one was a 2 percent nickel boiler steel, RR Specification 3-A; one was a 5 percent nickel steel, SAE Specification 2515; two were high-tensile-strength chromium steels, a proprietary of 3 percent chromium content and an AISI Type 501 D of 5 percent chromium content; and two were Federal Specification QQ-S-741 low-carbon steels, one copper-bearing and the other mild.

Immersion samples were 9 inches square and ¼ inch thick. Before exposure all were solvent cleaned and vapor degreased in trichloroethylene. They were then pickled at 170 F ± 5 F in 10 percent sulfuric acid for 10 minutes, with the exception of the mild steel samples that were to be exposed with a machined or an "as received" surface.

Immediately after pickling, the steels were rinsed in water and brushed with a vegetable fiber brush. This was followed by immersion for ½ to 1 hour in 170 F water to expel absorbed hydrogen. The test pieces were then dried in an oven at 160 F before being placed in a desiccator for cooling prior to initial weighing. All samples were accurately weighed, measured, inspected, and photographed before being installed in racks as shown in Figure 2. Panels were in contact only with ceramic insulators and were so oriented that the direction of rolling was horizontal.

Each rack contained four sets of duplicate specimens with glass barriers between each set and between the terminal specimens and the ends of the racks. This arrangement provided insulation against stray currents. The racks were located so that no two racks containing significantly dissimilar metals would be in proximate locations. Each pier position supported an upper and lower rack. In sea water the two levels were 14 ft apart, the lower rack being located 14 ft below mean tide for continuous exposure, and the top rack at mean tide elevation as shown in Figure 3. At the fresh water immersion site, shown in Figure 4, the two racks were submerged continuously.

Immediately upon removal, all samples were cleaned of marine growth and heavy corrosion product at the exposure sites. Following transference to the laboratory they were cleaned chemically by immersion in 10 percent sulfuric acid with 1 percent quinoline ethiodide inhibitor. Additional mechanical cleaning was employed as required. The cleaning loss of each sample was less than 40 mg/sq dm. These losses for the steels were not large enough to warrant consideration. Cleaned samples were weighed, measured, inspected, and photographed. Pit depths were referred to the original surface of the metal either

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He also has held offices in the American Society for Testing Materials. Other technical societies of which he is a member are the Federation of Paint and Varnish Production Clubs, the American Institute of Chemists, Washington Academy of Sciences, and Research Society of America (NRL Branch). He is presently an executive in the chemistry branch of the U. S. Naval Research Laboratory and was educated at the University of North Carolina with BS, MS and PhD degrees.

by measurement from an uncorroded surface or by calculation using the original and final average measured thickness of the sample. The average of the twenty deepest pits represents the average of the five deepest pits measured on each side of duplicate specimens (total area 2.25 sq ft). The methods of testing and exposure were reported in more detail in Part 1 of this series.[2]

Corrosion Results

A comprehensive evaluation of the results of exposure of the ten structural steels studied is presented as the average of duplicate panels in Table 3. The average penetration values were obtained by calculation from the average weight loss of the duplicate panels. These values indicate the general progression of corrosion over the total surface for each exposure period. Accelerated pitting is considered more significant than the average weight loss in certain types of construction. As an example, pitting on vessels, buoys and hulls, if permitted to proceed to perforation, is obviously more critical than larger weight losses spread evenly over entire surface areas. The average pitting attack on the structural steels tested is tabulated in Table 3 as the average twenty deepest pits. Also, the deepest single penetration on any of the four surfaces is given, but this is a less reliable value for all specimens because of the greater influence of chance and area exposed.

The pitting factor is included because of its general acceptance and utility in evaluating corrosion effects. Its accuracy is limited, however, since it is directly related to the value of the deepest pit.

Types of corrosion observed during inspection of cleaned panels were noted and are tabulated in the last column of Table 3. These corrosion types were described in detail with typical photographs in the first report of this series.[2]

Evaluation of the data in Table 3 indicates that for corrosion resistance in the tropical underwater environments of the Canal Zone, the unalloyed structural steel was in most cases equal to or superior to the steels containing small percentages of alloying constituents. The exceptions were in fresh water where advantages favoring some of the alloy steels containing chromium were indicated. Above half-tide, comparative corrosion resistance would probably increasingly favor the low-alloy steels, since in tropical marine atmospheric exposure there is a definite advantage for these steels.[3] In the fresh water of Gatun Lake the chromium steels were definitely superior to the unalloyed carbon steel as measured by weight loss and they were equal in resistance to pitting. However, for long-term exposure in sea water the steels containing chromium, surprisingly, were inferior to the unalloyed carbon steel.

Relative Intensities of Underwater Corrosion

The severity of corrosion of low-carbon unalloyed steels in the atmosphere varies from location to location according to the type and amount of impurities in the air, the amount of moisture present, and the average ambient temperature.[3,6-8] Similarly, variations in corrosion intensity may be expected for steel in different bodies of fresh waters. Fresh waters may differ in their properties and compositions to such an extent that results reported for their corrosion effect must be delineated for the specific prevailing condition. In a previous report in this series[2] a comparison of the corrosion of a low-carbon steel exposed in Gatun Lake, Canal Zone, and in the Allegheny, Monongahela, and Mississippi Rivers showed the corrosion rate at Gatun Lake to be in excess of each of the three river exposures, even though the pH of the Monongahela River was between 3.5 and 4.0. The increased corrosion rate in the Canal Zone probably can be attributed primarily to the higher average annual temperature of Gatun Lake (Figure 1) since the pH of the Gatun Lake water varies only between 7.0 and 8.0, and the water is relatively free of corrosive impurities as is shown by the chemical composition in Table 1.

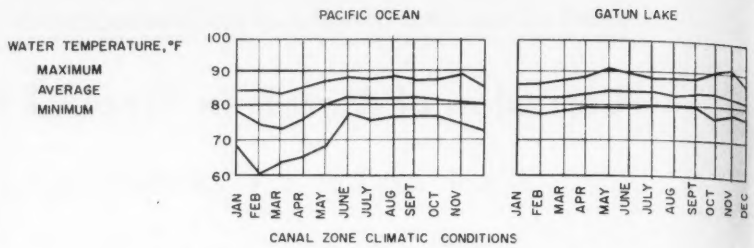


Figure 1—Water temperatures at the immersion test sites.

Open sea water is quite uniform in its composition throughout the oceans,[4] and as a result the corrosion rate of steels exposed in clean sea water should be comparable regardless of the locations. Differences in corrosion rates observed at different locations can then be attributed to variations in marine fouling, differences in water contamination near the shore lines, variations in velocity, and differences in water temperature.

A comparison of the corrosion rates of a low-carbon unalloyed steel exposed in the Pacific Ocean at Fort Amador, Canal Zone, on the east coast of the United States at Kure Beach, North Carolina, and on the west coast of the United States at Port Hueneme, California, is presented in Figure 5. The corrosion curve from the Kure Beach tests[9] almost parallels the corrosion curve from Fort Amador. However, the curve from the Port Hueneme tests[10] shows a considerably higher corrosion loss in 2½ years than do either of the curves from Kure Beach or Fort Amador. Since the average yearly temperature of the ocean water in the Canal Zone is approximately 20 F warmer than at Port Hueneme and 10 F warmer than at Kure Beach, this trend was unexpected. The deviation probably is due either to a greater marine organism activity in the tropical waters, providing a corrosion stifling effect, or to some accelerating factor in the temperate sea water tests, such as harbor contamination or water velocity. The average

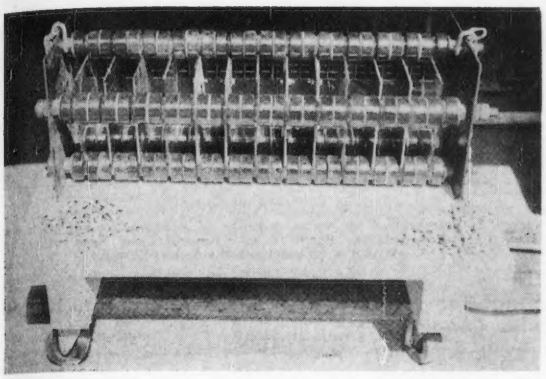


Figure 2—Typical immersion rack with specimens and glass barriers in place.

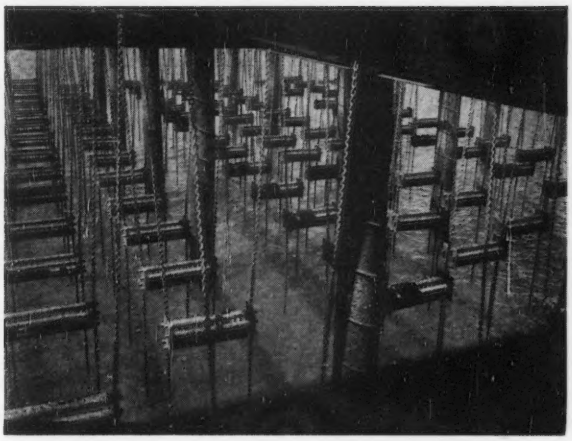


Figure 3—Sea water immersion specimens in place at Fort Amador (view at low tide).

TABLE 1—Summary of Individual Analyses of Water Samples Obtained at the Immersion Test Sites

Constituent or Property Determined	Fresh Water, Gatun Lake at Gatun			Sea Water, Pacific Ocean at Fort Amador					
				Upper Rack Level Elevation 0.0 ft.			Lower Rack Level Elevation–14.0 ft.		
	Maximum	Minimum	Average	Maximum	Minimum	Average	Maximum	Minimum	Average
Electrical Conductivity (mhos x 10^{-3} at 81 F)	0.12	0.091	0.11	51.7	21.3	42.2	51.7	35.4	45.4
Total Dissolved Solids (ppm)	165	69	113	42,776	22,613	35,832	41,480	26,390	35,735
Total Suspended Solids (ppm)	23	0.0	7.6	220	0	64	173	0	49
Turbidity (ppm)	<5	<5	<5	20	<5	<5	25	<5	<5
Oxygen Saturation (percent)	98	78	90	105	62	90	103	64	87
Oxygen Consumed (ppm)	2.4	0.7	1.4	2.5	0.6	1.6	2.6	0.4	1.6
Biochemical Oxygen Demand (ppm)	2.2	0.1	1.0	3.4	0.2	1.6	2.3	0.0	1.5
pH (colorimetic)	8.0	6.9	7.5	8.4	7.8	8.2	8.4	7.8	8.1
Organic and Volatile Matter (ppm)	65	6.6	34	10,379	2,150	6,226	10,632	2,759	6,236
Sulfate (ppm)	7.2	0.0	2.6	3,240	1,590	2,431	3,177	1,837	2,437
Chloride (ppm)	12.5	0.0	7.0	20,098	11,300	17,415	19,949	10,379	17,357
Nitrate (ppm)	Trace	0.0	Trace	0.01	0.00	Trace	0.01	0.00	Trace

TABLE 2—Composition of Structural Steels Exposed in Tropical Waters

Steel	Type	Specification	Composition (Percent)								
			C	Mn	P	S	Si	Cr	Ni	Cu	Mo
A	Unalloyed Low Carbon	QQ-S-741, Type 11 Grade A, Class 1	0.24	0.48	0.040	0.027	0.008	0.03	0.051	0.080	....
D	Copper-Bearing	QQ-S-741, Type 11 Grade A, Class 2	0.22	0.44	0.019	0.033	0.009	Trace	0.14	0.35	....
E	Nickel (2%)	RR-SPECS-3-A	0.20	0.54	0.012	0.023	0.18	0.15	1.94	0.63	....
F	Nickel (5%)	SAE-2515 5% Ni Steel	0.13	0.49	0.010	0.014	0.16	0.10	5.51	0.062	....
G	Chromium (3%)	Max. 0.10% C Hot Rolled	0.08	0.44	0.010	0.017	0.13	3.16	0.16	0.11	0.02
H	Chromium (5%)	AISI Type 501D	0.08	0.41	0.020	0.019	0.20	5.06	0.11	0.062	0.52
I	Low-Alloy	Proprietary Cu-Ni	0.08	0.47	0.007	0.026	0.060	None	1.54	0.87	....
J	Low-Alloy	Proprietary Cu-Cr-Si	0.15	0.45	0.113	0.026	0.47	0.68	0.49	0.42	....
K	Low-Alloy	Proprietary Cu-Ni-Mn-Mo	0.078	0.75	0.058	0.022	0.04	Trace	0.72	0.61	0.13
L	Low-Alloy	Proprietary Cr-Ni-Mn	0.13	0.60	0.089	0.021	0.15	0.55	0.30	0.61	0.059

composition of the sea water at Fort Amador is given in Table 1 and monthly temperature averages of the water are shown in Figure 1.

Sea water corrosion in the tropics is undoubtedly affected by marine fouling. The 80 F average water temperature of the Pacific Ocean at Panama affords a continuous growing season for marine organisms; the attachment of fouling to test panels in these tests was profuse both in continuous immersion and at mean tide. On steel panels this growth probably offered some protection from corrosion. The presence of these organisms and their unmeasurable effect on the corrosion of metals must explain, at least in part, the unusual shape of some of the corrosion curves presented. This fouling effect punctuates one of the defects of using short-term or synthetic-water tests for establishing the life of ferrous metals in natural sea water.

The corrosion rates of unalloyed low-carbon steel at the three Canal Zone exposure sites are presented in Figure 6. An outstanding characteristic of these curves is the fact that after eight years

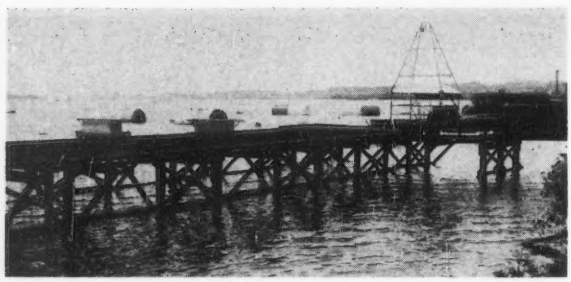


Figure 4—General view of pier for fresh water immersion at Gatun Lake.

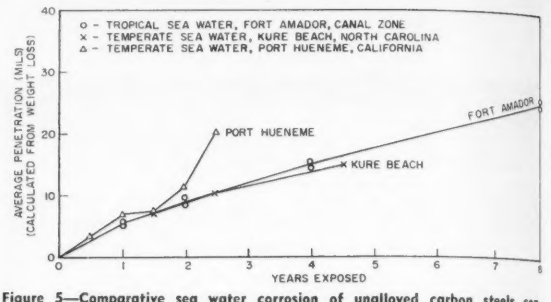


Figure 5—Comparative sea water corrosion of unalloyed carbon steels continuously immersed in the Pacific Ocean, Panama Canal Zone, and on the east and west coasts of the United States.

TABLE 3—Comprehensive Evaluation of Corrosion Damage for Ten Structural Steels Exposed to Natural Waters in the Panama Canal Zone

Steel	Exposure	Weight Loss (g/dm²)				Average Penetration (Mils)*				Average 20 Deepest Pits (Mils)**				Deepest Pit (Mils)**				Pitting Factor***				Corrosion Type****
		1 yr.	2 yr.	4 yr.	8 yr.	1 yr.	2 yr.	4 yr.	8 yr.	1 yr.	2 yr.	4 yr.	8 yr.	1 yr.	2 yr.	4 yr.	8 yr.	1 yr.	2 yr.	4 yr.	8 yr.	8 yr.
D	Sea Water Immersion	11.76	18.26	29.10	55.29	5.9	9.2	14.6	27.7	36	44	56	63	61	121	120	108	10	13	8	4	RAQH
	Sea Water Mean Tide	22.43	22.55	22.98	48.26	11.3	11.3	11.5	24.2	24	18(18)	7(2)	45	38	28	10	63	3	2	1	3	AR
	Lake Water Immersion	15.66	24.24	35.18	46.36	7.9	12.2	17.6	23.3	21	35	48	63	32	47	75	82	4	4	4	4	EF
E	Sea Water Immersion	14.79	18.29	34.08	56.04	7.5	9.2	17.2	31.7	32	51	50	94	51	86	84	179	7	9	5	6	ARHQF
	Sea Water Mean Tide	17.70	17.88	27.52	45.58	8.9	9.0	13.9	22.9	20	..(0)	22	39	32	...	67	50	4	..	5	2	AFR
	Lake Water Immersion	14.71	25.31	34.69	40.18	7.4	12.7	17.4	20.2	19	40	43	64	29	54	62	89	4	4	4	4	EQF
F	Sea Water Immersion	12.50	20.05	33.20	63.68	6.3	10.1	16.7	32.0	30"18)	50	77	117	57	84	195	214	9	8	12	7	RAHQ
	Sea Water Mean Tide	15.56	17.23	25.44	39.82	7.8	8.7	12.8	20.0	..(0)	..(0)	30(19)	39	...	...	64	75	..	..	5	4	AR
	Lake Water Immersion	13.11	24.57	35.52	46.78	6.6	12.3	17.8	23.5	19(19)	44	58	69	33	55	77	100	5	4	4	4	EQ
G	Sea Water Immersion	4.13	10.92	35.61	80.44	2.1	5.5	18.0	40.5	16(10)	31	39	65	20	38	49	78	10	7	3	2	R
	Sea Water Mean Tide	22.11	23.56	29.05	51.08	11.2	11.9	14.7	25.7	23	24	56	82	32	45	72	93	3	4	5	4	DR
	Lake Water Immersion	4.84	7.29	10.79	19.30	2.4	3.7	5.5	9.7	24	27	32	54	28	36	38	63	12	10	7	6	RAQH
H	Sea Water Immersion	5.34	12.13	33.54	63.49	2.7	6.1	17.0	32.0	27	29	42	63	91	50	51	90	34	8	3	3	R
	Sea Water Mean Tide	23.32	22.63	24.85	48.59	11.8	11.4	12.5	24.5	30(18)	41	70	88	37	45	81	99	3	4	6	4	DR
	Lake Water Immersion	3.80	4.49	7.10	17.09	1.9	2.3	3.6	8.7	23	29	35	52	29	35	42	58	15	15	12	7	RAK
I	Sea Water Immersion	11.66	16.88	41.23	52.64	5.8	8.5	20.7	26.4	54	62	37(19)	82	85	89	68	152	15	10	3	6	RAHQ
	Sea Water Mean Tide	23.68	26.00	29.95	79.29	11.9	13.1	15.0	39.7	..(0)	..(0)	84	70	...	...	185	134	..	..	12	3	RA
	Lake Water Immersion	12.07	19.67	26.04	32.62	6.1	9.9	13.1	16.4	18(19)	33	43	68	31	55	74	85	5	6	6	5	R
J	Sea Water Immersion	13.62	22.27	27.69	85.70	6.9	11.2	14.0	43.2	31	50	61	80	46	79	79	175	7	7	6	4	RAHF
	Sea Water Mean Tide	21.44	19.49	24.16	41.89	10.8	9.8	12.2	21.1	20	..(0)	30	47	27	...	43	54	3	..	4	3	E
	Lake Water Immersion	11.33	17.17	23.56	33.11	5.7	8.7	11.9	16.7	40	55	63	79	56	63	89	93	10	7	7	6	EQ
K	Sea Water Immersion	12.80	19.06	22.97	50.85	6.5	9.6	11.5	25.5	23	37	36	56	44	105	81	139	7	11	7	5	ARHQLF
	Sea Water Mean Tide	21.61	30.33	37.30	49.34	10.8	15.2	18.7	24.8	17(19)	36	34	40	23	69	43	94	2	5	2	4	RALF
	Lake Water Immersion	14.50	22.57	31.73	44.62	7.3	11.3	15.9	22.4	24	43	55	81	33	55	81	120	5	5	5	5	RF
L	Sea Water Immersion	12.57	21.93	41.56	87.35	6.3	11.0	20.9	43.9	26	47	64	97	36	76	118	259(p)	6	7	6	6	RH
	Sea Water Mean Tide	18.17	17.97	21.53	40.86	9.1	9.0	10.8	20.5	16	..(0)	24	39	22	...	31	50	2	..	3	2	RA
	Lake Water Immersion	10.68	16.27	25.31	31.08	5.4	8.2	12.8	15.6	36	67	82	112	47	85	111	146	9	10	9	9	RQ
A	Sea Water Immersion	11.84	16.55	31.39	50.88	6.0	8.3	15.8	25.5	41	47	66	66	62	61	113	86	10	7	7	3	RAHQ
	Sea Water Mean Tide	18.85	18.51	25.39	46.30	9.5	9.3	12.5	23.2	18(8)	16(7)	27(19)	40	27	23	55	65	3	2	4	3	ERH
	Lake Water Immersion	15.22	23.72	33.95	43.68	7.8	12.2	17.0	21.9	20	38	45	58	24	46	66	71	3	4	4	3	E
A (Mill-scale)	Sea Water Immersion	8.38	15.53	30.09	46.49	4.2	7.8	15.1	23.4	55	77	108	168	104	113	158	263(p)	25	14	10	11	QKHF
	Sea Water Mean Tide	18.39	22.48	26.28	36.85	9.3	11.3	13.2	18.5	16	18	23	43	21	30	40	75	2	3	3	4	E
	Lake Water Immersion	12.58	18.99	27.23	37.67	6.3	9.6	13.7	18.9	29	43	34	48	40	67	51	74	6	7	4	4	EK
A (Ma-chined)	Sea Water Immersion	11.01	18.04	29.81	53.93	5.5	9.1	15.0	27.1	38	61	57	58	64	157	118	157	12	17	8	6	RAQH
	Sea Water Mean Tide	21.05	21.73	30.51	49.52	0.6	10.9	15.3	24.9	21(18)	21(15)	35(15)	58	40	29	57	124	4	3	4	5	RAQ
	Lake Water Immersion	15.02	23.21	31.75	40.25	7.5	11.7	16.0	20.2	22	37	53	61	26	47	62	88	3	4	4	4	EH

* Calculated from weight loss and specific gravity.

** Pit depths referred to the original surface of the metal either by measurement from an uncorroded surface or by calculation using the original and final average measured thickness of the sample. Average of 20 deepest pits represents average of the 5 deepest pits measured on each side of duplicate specimens (area, 2.25 sq. ft.); values in parentheses indicate total number averaged when less than 20. Perforation of plate by deepest pit is indicated by (p).

*** Ratio of the deepest measured penetration to the average calculated penetration.

**** All corrosion types are described in detail in Part 1 of this report series. Those referred to here are: A—Uniform Attack. D—Pitting Attack (Uniform). E—Localized Attack Uniform), F—Stress, H—Concentration Cell, K—No Visible Attack, L—Defect in Metal, Q—Pitting Attack (Random), and R—Localized Attack (Random).

a corrosion difference of no more than 15 percent exists between the sea water, fresh water, and mean tide exposures. At four years, the corrosion of steel in fresh water was slightly higher than in sea water, but the curves cross at about 5½ years and as the fresh water rate continued to decrease, the sea water rate remained relatively linear. During the first year the corrosion rate at mean tide was higher than for either of the immersed conditions. This rate then flattened out considerably between one and four years to a point where it was the least severe. However, an increase was noted again between four and eight years, and at eight years the slope was steeper than for continuous sea water immersion. It would appear that at some time between installation and one year the original high rate of corrosion was slowed perceptibly by the combination of protective corrosion product buildup and tightly adhering heavy marine organism attachment that developed at this location. This same type of corrosion occurred to an even more pronounced degree on most of the other steels exposed at mean tide. A possible explanation for this behavior may be that after four years, the hitherto protective layers of attached fouling and corrosion product became so heavy that both were scaled off and a new or more poorly protected surface was exposed to renewed attack.

Several investigators have reported previously that mean tide exposure is generally more corrosive than continuous sea water immersion, but in this investigation, for exposure periods of more than two years, the reverse effect was found. Of all the structural steels tested, only one, steel I, was corroded more

severely at the mean tide location than in continuous sea water exposure at the end of eight years. The corrosion rate at mean tide for the first year was appreciably higher than the rate for continuous sea water exposure. However, after two years, this rate, as previously described, slowed perceptibly. After eight years, the steels at mean tide, with the exception of steel I, averaged approximately 35 percent lower corrosion rate than those exposed to continuous immersion in sea water.

The average pitting attack also was less severe at mean tide than during continuous immersion in either sea or fresh water. Exceptions were the 3 percent and 5 percent chromium steels which were most severely pitted at mean tide. Cross-sectional photographs of panels of mild unalloyed structural steel after eight years at each of the exposure sites are shown in Figure 7.

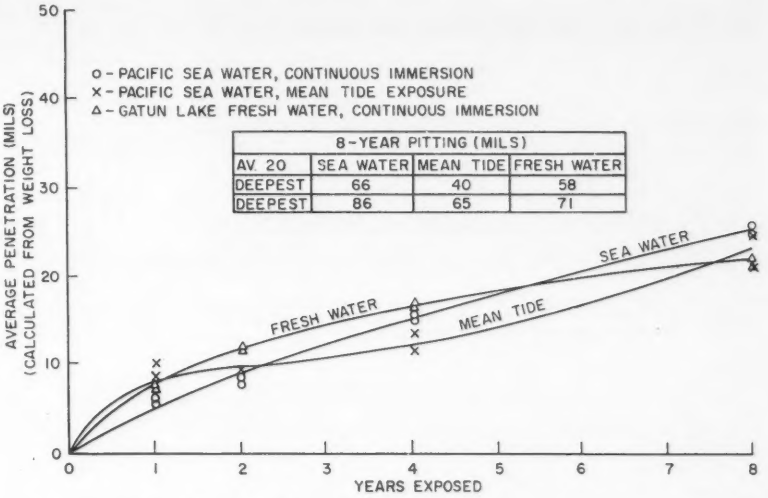


Figure 6—Comparison of corrosion of unalloyed low-carbon steel continuously immersed in sea water, in fresh water and exposed at mean tide elevation in the Panama Canal Zone.

Effect of Surface Conditions

Millscale on unalloyed low-carbon steel which is continuously immersed in sea water will unquestionably intensify localized corrosion. This is due to the cathodic behavior of the millscale in relation to the underlying steel. However, since quantitative information on the type and intensity of attack in tropical waters is meager, it was decided to expose the steel to each of the exposure conditions in these tests with millscale, pickled, and machined surfaces. The bar diagrams in Figure 8 show the results of exposing these three surface conditions to the three types of immersion.

As expected, the pitting attack on the millscale panels proceeded during continuous immersion in sea water to a point where, at the end of eight years, there were three perforations of 0.263-inch-thick plate. The average of the twenty deepest pits for this exposure condition was almost three times the average for the panels with the pickled and machined surfaces. Pitting attack on the pickled surfaces was somewhat higher than on the machined surface, but the ratio of weight loss between the three surface conditions was reversed with reference to pitting; i.e., the panels with the millscale surface lost the least weight, and those with machined surface the most. Accelerated local attack was not in evidence at the mean tide location or in fresh water. The millscale surfaces lost the least weight in both of these locations and the average of the twenty deepest pits on the millscale surface was the lowest in fresh water and lower than the machined surface in the mean tide location; however, it was slightly higher than the pickled surface at this same location.

The change in temperature of the panels at mean tide, due to their alternate wetting and drying, probably facilitated the loosening of the millscale at an early stage thereby permitting the panels to corrode in the same manner as did the steel with a descaled surface. This fact, plus the fact that the panels were deprived of a continuous sea water electrolyte, no doubt accounts for the absence of any significant effect from a millscale surface at the mean tide location. The conductivity of the fresh water was sufficiently low that the establishment of an effective galvanic cell between the millscale and bare areas on the plate did not occur.

Surface photographs of the millscale and pickled surfaces from the sea water immersion are shown in Figure 9. Figure 10 shows cross-sectional photographs from each of the exposures after eight years.

Effect of Low-Alloy Additions

Copper-Bearing Steel. Alloying steel with small amounts of copper in quantities of 0.1 percent to 0.5 percent has been found effective in reducing atmospheric corrosion.[3,8] On the other hand, LaQue[5] and Speller[11] report that the inclusion of low percentages of copper seems to have no measurable influence on the corrosion rates of steel immersed in sea water. The question was raised as to whether this would hold true for the mean tide exposure in Panama where the specimens were out of water and above the splash zone for long periods each day. Furthermore, would these conclusions[5,11] based on tests in sea water, hold also for steel immersed in the warm fresh water of Gatun Lake? To answer these questions the investigation of steel containing small amounts of copper appeared warranted.

The copper steel included in the investigation contained 0.35 percent copper. Results from the eight-year exposure are compared with results with mild unalloyed steel in Figure 11. The time-corrosion curves show the progression of weight loss penetration for each immersion. The pitting penetration after eight years is tabulated with each curve. Examination of these curves shows that no significant difference in the corrosion rates of the two steels exists. The differences are so small that a curve drawn through the average of the four points from both steels at each time period probably would be more representative of the true shape of the corrosion curve for either metal.

The shapes of the curves obtained for these two steels are typical of most of the steels investigated in these tropical waters. The curves for continuous sea water exposure approach a linear relationship with time after two years and the slope of the straight-line portion indicates a corrosion rate of approximately 3 mils/year. The checks between duplicate panels continuously immersed in sea water are generally very good.

Panels exposed at mean tide, as mentioned previously, corroded very rapidly for the first year, but between one and four years weight loss was insignificant. This was followed by an acceleration in corrosion between four and eight years and at eight years the rate, as determined by the slope of the curves, was equal to 4 to 5 mils/year. The results from duplicate specimens were more divergent in the tide range area than in any other. Of additional interest is the fact that marine atmospheric corrosion curves for these same two steels were of the same general shape as the mean tide curves, but with leveling off occurring between two and four years.[3]

Steels immersed in fresh water gave the most even weight loss of any exposure, and checks between duplicate panels were excellent for every period. The two curves in Figure 11 (fresh water) with constantly decreasing slopes indicate a steady inhibition of corrosion by corrosion product. The rate of attack shown by the slope of the curves, decreases from 7.5 mils/year at the end of one year to only about 1 mil/year at eight years.

Nickel Steels. Low-alloy nickel steel has been in use in Canal Zone waters in components of the Panama Canal lock structures for many years. In no instance, however, has there been an evaluation of corrosion resistance on a controlled basis, and it could not be ascertained definitely whether increased corrosion re-

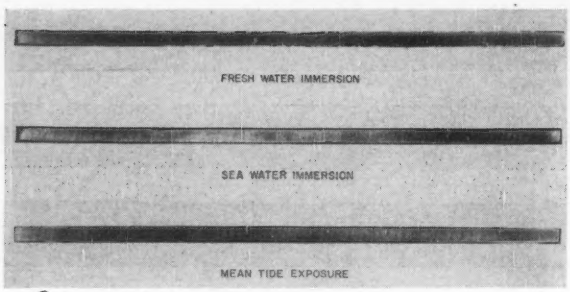


Figure 7—Comparison of typical corrosion damage to low-carbon unalloyed structural steel exposed for eight years to three Canal Zone underwater environments (cross-sectional photographs, approx. ½X).

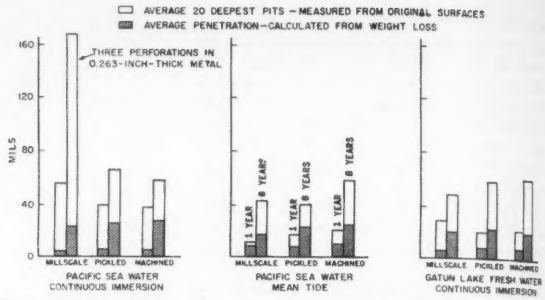


Figure 8—Comparison of effect of millscale, pickled and machined surfaces on the corrosion of low-carbon unalloyed steel exposed in the tropical waters of the Panama Canal Zone. Left bar is for 1 year; right bar is for 8 years.

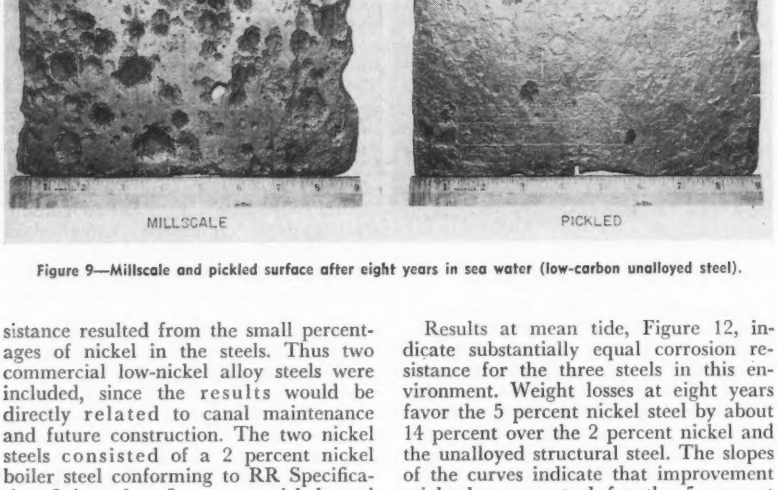


Figure 9—Millscale and pickled surface after eight years in sea water (low-carbon unalloyed steel).

sistance resulted from the small percentages of nickel in the steels. Thus two commercial low-nickel alloy steels were included, since the results would be directly related to canal maintenance and future construction. The two nickel steels consisted of a 2 percent nickel boiler steel conforming to RR Specification 3-A and a 5 percent nickel steel (steels E and F, Table 2), designated as SAE-2515.

As shown in Figure 12, nickel steels immersed continuously in tropical sea water for eight years are definitely inferior to mild structural steel on the basis of both general penetration and local attack. The 3 percent and 5 percent nickel steels showed identical weight loss values during this exposure and at the end of eight years these losses were 25 percent higher than those for mild steel. The slope of the curves between two and eight years indicates a constant corrosion rate of 3.6 mils/year versus 2.7 mils/year for the unalloyed steel. As may be seen by examination of the cross-sectional photographs in Figure 13 and the eight-year pitting information given for continuous sea water immersion in Figure 12, a less favorable position for nickel steel is revealed. The averages of the twenty deepest pits on the 3 percent and 5 percent nickel steels were 42 percent and 76 percent deeper, respectively, than the average of the twenty deepest pits on the unalloyed mild steel.

Results at mean tide, Figure 12, indicate substantially equal corrosion resistance for the three steels in this environment. Weight losses at eight years favor the 5 percent nickel steel by about 14 percent over the 2 percent nickel and the unalloyed structural steel. The slopes of the curves indicate that improvement might be expected for the 5 percent nickel steel for longer exposure periods. Pitting of the three metals in this environment was practically equal.

In Gatun Lake also, no appreciable advantage was apparent for the nickel alloys. At eight years the weight loss of the 5 percent nickel steel was slightly higher and the 2 percent slightly lower than the unalloyed structural steel, Figure 12. Pit depths increased slightly with increasing nickel content.

Chromium Steels. The results from the steels containing chromium as an alloying element were among the most interesting of the entire series. It has been determined previously by several investigators[12-15] that the inclusion of small percentages of chromium in steel provides significant improvement in the corrosion resistance in sea water; LaQue[5] states that 3 percent chromium is the probable optimum for sea water exposure. Two chromium steels were included in the present tests, a proprietary metal containing 3 percent chromium, and an AISI Type 501D alloy of 5 percent chromium (steels G and H in Table 2). In addition two of the proprietary low-alloy steels (J and L in Table 2) contained small percentages of chromium as an alloying constituent, 0.68 percent chromium in steel J and 0.55 percent chromium in steel L. For the first year of sea water immersion both the 3 percent and 5 percent chromium steels were appreciably more resistant to corrosion than was the unalloyed mild steel, Figure 14. Although still somewhat more resistant after two years, the curves for both chromium steels crossed the mild steel curve at about 3½ years. At the end of eight years the loss in weight of the 5 percent chromium steel exceeded that of the mild steel by 30 percent while the 3 percent chromium steel had lost 63 percent more weight than had the mild steel. The low-alloy steels (steels J and L) containing smaller percentages of chromium also exhibited this high rate of corrosion during the later stages of exposure.

The extent of localized attack in continuous sea water immersion can be evaluated from the cross-sectional views of the three metals shown in Figure 15 and from the eight-year pitting values given with each group of curves in Figure 14. The depth of pitting or localized attack was practically equal for these three steels immersed in sea water but the frequency of pitting was much higher on the chromium steels, and coincident attack from two sides probably would result in perforation of the panels at an earlier stage.

During exposure to sea water at mean tide a somewhat different corrosion effect was noted. In this environment the pitting of both chromium steels was quite severe whereas the unalloyed steel showed a uniform and mild attack as shown in the cross-sectional photographs in Figure 16 and the pitting penetration values in Figure 14. In addition to the deep pits in the chromium steels which show plainly in the photographs, the entire remaining surface (Figure 17) was covered by minute pits giving the panels a unique lacey appearance. This same distinctive corrosion pattern, but to a lesser degree, was observed on the low-alloy steels containing 0.55 percent and 0.68 percent chromium. Only on the steels in which chromium was an alloying element was the effect apparent.

Average penetration curves for mean tide exposure (Figure 14) indicate, that in spite of the much deeper pitting, there

was only a slight increase in weight loss of the two chromium steels over the mild steel, and the same typical curve shapes were obtained for all.

In the fresh water of Gatun Lake the chromium steels appeared to their best advantage and for eight years they showed the lowest average penetration of any of the steels tested in fresh water. Their eight-year loss in weight was only about 40 percent of that for mild unalloyed steel (Figure 14) and they were equally superior in this respect at all shorter periods. The slight upward trend of the curves does not seem significant at this time, but in view of the results obtained in sea water it does seem possible that the upward trend may increase and eventually cross the mild steel curve. Pitting at eight years was approximately equal in magnitude for the 3 percent chromium, 5 percent chromium, and unalloyed steel. Figure 14 shows the comparative pitting values and in Figure 18 are photographs of typical cross sections after eight years' exposure in Gatun Lake waters.

Because of the uneven corrosion of the chromium steels a variation in weight loss between duplicate panels might be expected, but did not occur. Checks between values for duplicate panels as shown in Figure 14 were excellent for both chromium steels in all three environments.

Proprietary Low-Alloy Steels. The atmospheric corrosion resistance of the proprietary low-alloy steels containing small amounts (usually less than 1 percent) of two or more alloying constituents has been well established by Copson,[8] Larrabee,[9] and Hudson and Stanners,[15] and confirmed in the tropical atmosphere tests in this investigation.[3] Similar results were not expected in tropical sea water immersion but it was considered desirable to determine exactly what these effects might be.

At mean tide, where the specimens are alternately immersed, and in the atmosphere as well as in the fresh waters of Gatun Lake, the effects of low-alloy additions were unpredictable. Four well-known brands of these low-alloy steels were included in the investigation, steels I, J, K, and L in Table 2. Figure 19 shows the corrosion-time curves for average calculated penetration along with a tabulation of the eight-year pitting results for the four steels in comparison with an unalloyed low-carbon steel.

In continuous sea water immersion, weight loss, as shown by Figure 19, indicates that after eight years' exposure the unalloyed steel was equal to the best of the proprietary low-alloy steels. The shapes of the curves show that one of the low alloys (steel I) will probably corrode least after longer periods of exposure. It should be pointed out, however, that the scattering between duplicate points for some of these metals in sea water is such that extrapolation of the curves may yield erroneous conclusions. The most severely corroded steels in this group (J and L), already discussed in the section on chromium steels, were those containing chromium as the

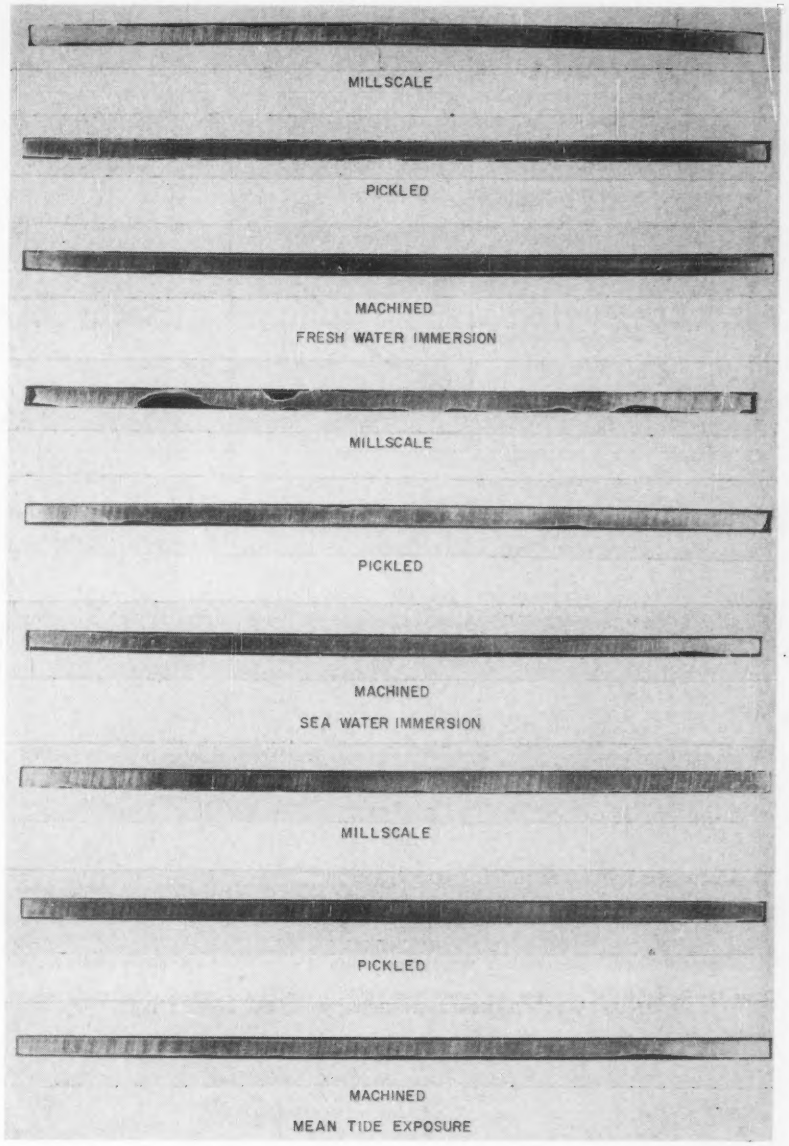


Figure 10—Comparison of eight-year corrosion attack on low-carbon unalloyed steel with millscale, pickled and machined surfaces (cross-sectional photographs, approx. 2/3X).

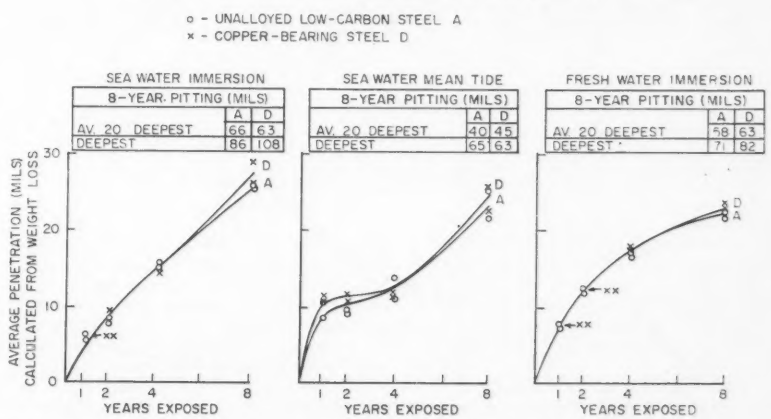


Figure 11—Comparative corrosion of copper-bearing and unalloyed low-carbon steels in the tropical waters of the Panama Canal Zone.

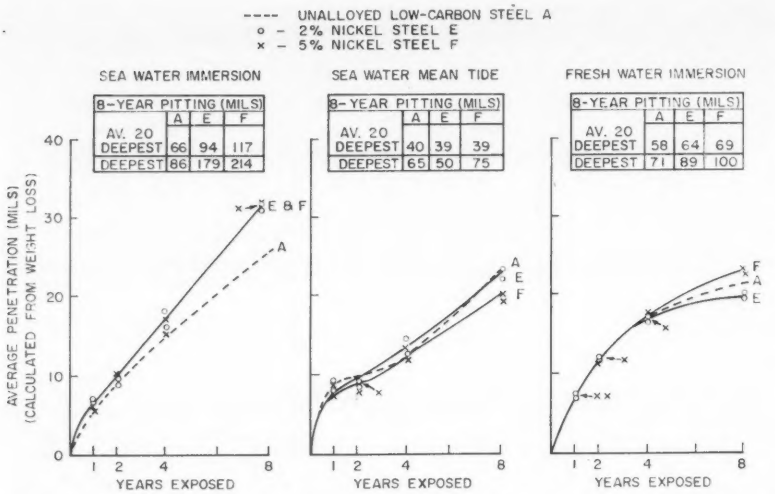


Figure 12—Comparative corrosion of nickel steels and unalloyed low-carbon steel in tropical waters of the Panama Canal Zone.

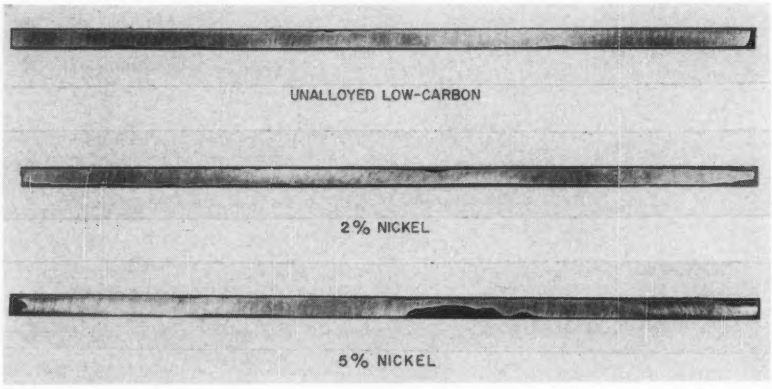


Figure 13—Comparison of unalloyed low-carbon steel with 2 percent and 5 percent nickel steels exposed for eight years in sea water (cross-sectional photographs, approx. 2/3X).

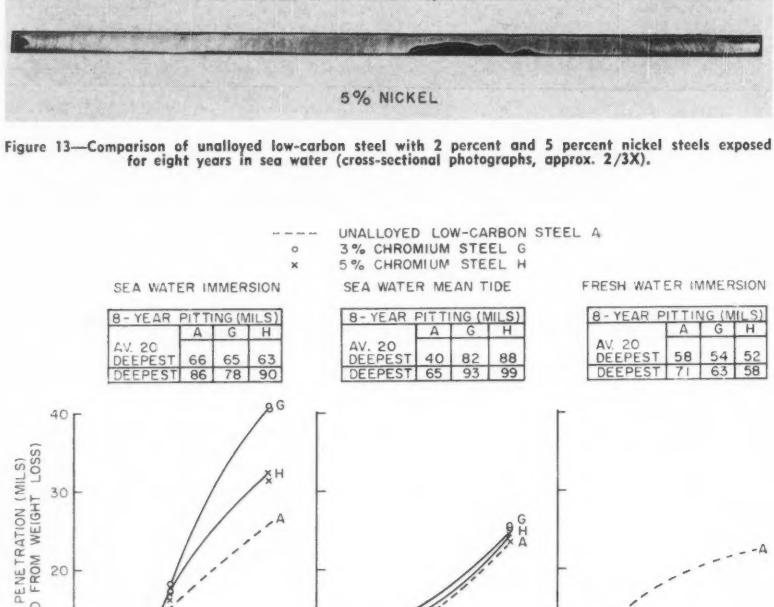


Figure 14—Comparative corrosion of chromium steels and unalloyed low-carbon steel in the tropical waters of the Panama Canal Zone.

major alloying constituent. Pitting after eight years' immersion in sea water was deeper on three of the four low-alloy steels than on the unalloyed carbon steel. Two of these three more severely pitted steels were those containing chromium (steels J and L). Considering the average of the twenty deepest pits, steel K was the best of the group and slightly superior to the unalloyed steel A. However, the single deepest penetration at eight years was appreciably deeper on all of the low-alloy steels than on the mild steel. Typical cross-sectional photographs of the most and least severely corroded proprietary low-alloy steels after eight years' exposure in sea water are compared with unalloyed structural steel in Figure 20.

Mean tide results revealed a reverse situation with the chromium-bearing steels (J and L). They were slightly more resistant than the mild steel to weight loss and equal in pitting. Steels K and I showed very peculiar results in this environment. Steel K was the most severely corroded of any of the group at two and four years, but corrosion slowed considerably between four and eight years. Furthermore, the direction of the curve indicates that this metal will be the least corroded of the four at some future date.

On all of the steels covered in this report, with the exception of steel K, the corrosion rate at the mean tide location was arrested between one and four years, causing a downward deflection in the corrosion-time curves. Steel I corroded more rapidly than most during the first four years of exposure but followed the normal trend for mean tide. Between four and eight years, however, the corrosion rate for this metal jumped amazingly to over 7 mils/year resulting in the steepest slope at eight years of any of the steels reported. Careful examination of the test data and exposed plates revealed no reason for this sudden increase. Figure 21 shows typical cross-sectional photographs of the least and most corroded steels at mean tide in comparison with the unalloyed carbon steel. Examination of these photographs and of the eight-year pitting data for mean tide exposure (Figure 19) indicates that unalloyed structural steel and three of the proprietary low-alloy steels were approximately equally resistant to pitting attack. However, metal I showed a much higher eight-year pitting penetration.

During fresh water immersion in Gatun Lake the low-alloy steels displayed some advantage over unalloyed mild steel. Three of the four lost only about 70 percent as much weight as unalloyed steel, and steel K was about equal to the unalloyed steel. Corresponding reductions in pitting did not occur. On the basis of pitting, the mild unalloyed steel was the best of the group. This may be seen in Figure 22 by comparing the cross-sectional photographs of the most severely and least severely corroded metals of the low-alloy group with the unalloyed steel.

It is strikingly demonstrated by this group of steels that an alloy which resists corrosion in one environment is not

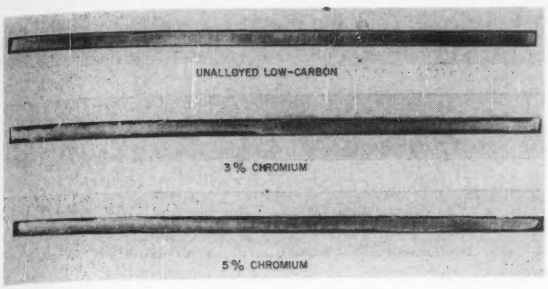


Figure 15—Comparison of unalloyed low-carbon steel with 3 percent and 5 percent chromium steels exposed for eight years in sea water (cross-sectional photographs, approx. ½X).

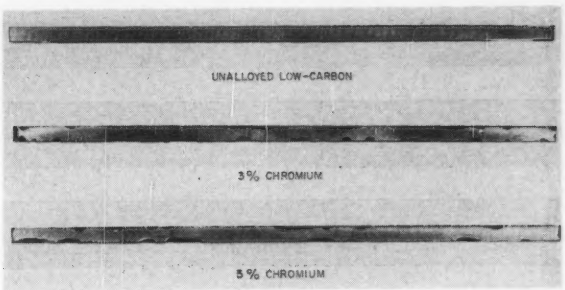


Figure 16—Comparison of unalloyed low-carbon steel with 3 percent and 5 percent chromium steels exposed for eight years to sea water at mean tide elevation (cross-sectional photographs, approx. ½X.)

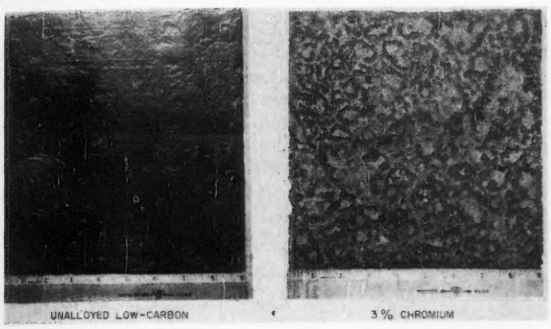


Figure 17—Unalloyed low-carbon and 3 percent chromium steels after eight years' exposure at mean tide.

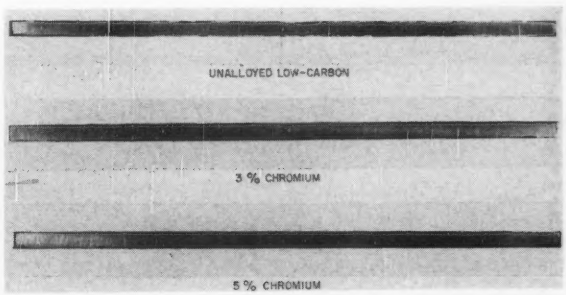


Figure 18—Comparison of unalloyed low-carbon steel with 3 percent and 5 percent chromium steels exposed for eight years in Gatun Lake fresh water (cross-sectional photographs, approx. ½X.)

necessarily the most resistant in some other condition of exposure. Those alloys which were superior at mean tide were often the poorest when continuously immersed in sea water. The composition of some of the low-alloy steels obviously detracted from their corrosion resistance in certain conditions of exposure. It is apparent also that mild structural steel would be a superior choice for variable immersion exposure conditions in the tropics.

Conclusions

1. The corrosion of unalloyed low-carbon steel in Panama tropical sea water is of the same magnitude as temperate sea water corrosion at Kure Beach, North Carolina, and less than corrosion at Port Hueneme, California. Because of the higher average water temperature in Panama it was expected that corrosion rates would be higher. The profuse marine growth on the panels in the Panama tests probably afforded some protection to the steel.

2. There is little difference in the corrosion rates of unalloyed low-carbon steel at the three environments in the Panama tests. At the end of eight years a maximum of 15 percent difference was noted between continuous immersion in sea water, in fresh water, and at mean tide exposure. However, the shape of the curves for these exposures indicates that sea water corrosion is proceeding at a linear rate, fresh water corrosion is steadily decreasing with time, and mean tide corrosion is accelerating.

3. Accelerated pitting attack on unalloyed low-carbon steel with a millscale surface was evidenced only in the sea water immersion exposure. Pitting attack and weight loss on millscale, pickled, and machined surfaces were essentially equal at the end of eight years in both the fresh water and mean tide exposures.

4. No significant difference in the type or magnitude of corrosion was found between copper-bearing steel and unalloyed low-carbon steel in sea and fresh water and at mean tide.

5. The inclusion of 2 percent and 5 percent of nickel in structural steel did not increase its corrosion resistance in fresh water or at mean tide. In sea water the nickel addition accelerated both weight loss and pitting-type corrosion attack.

6. Addition of chromium in the amounts of 3 percent and 5 percent in structural steel was beneficial in imparting corrosion resistance to panels im-

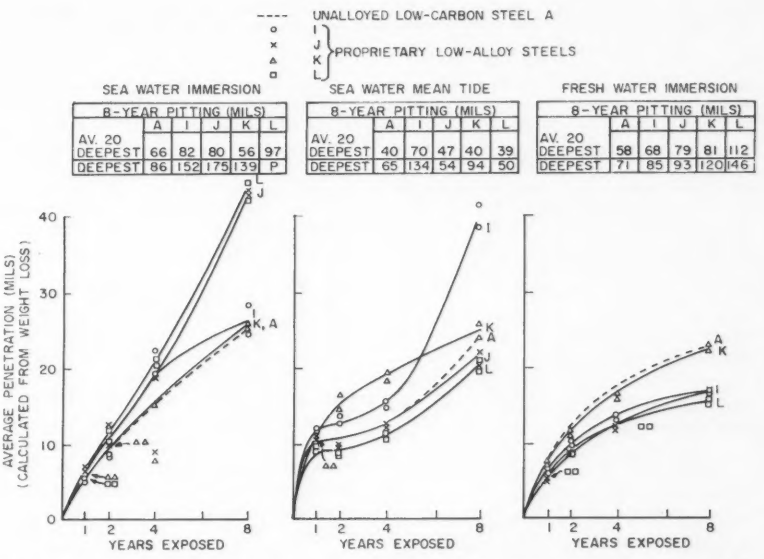


Figure 19—Comparative corrosion of a group of proprietary low alloy steels and an unalloyed low-carbon steel in the tropical waters of the Panama Canal Zone.

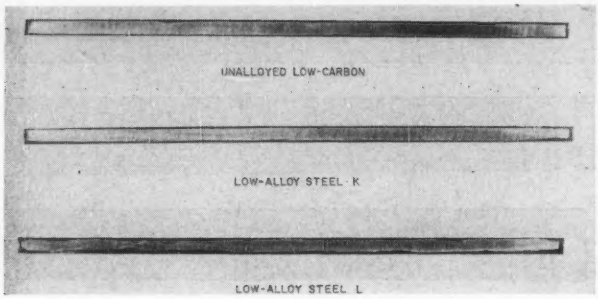


Figure 20—Comparison of the most and least corroded low-alloy steel with unalloyed low-carbon steel, exposed in sea water for eight years (cross-sectional photographs, approx. ½X).

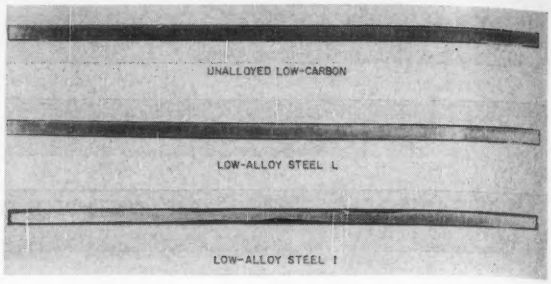


Figure 21—Comparison of the most and the least corroded low-alloy steel with unalloyed low-carbon steel, exposed at mean tide for eight years (cross-sectional photographs, approx. ½X).

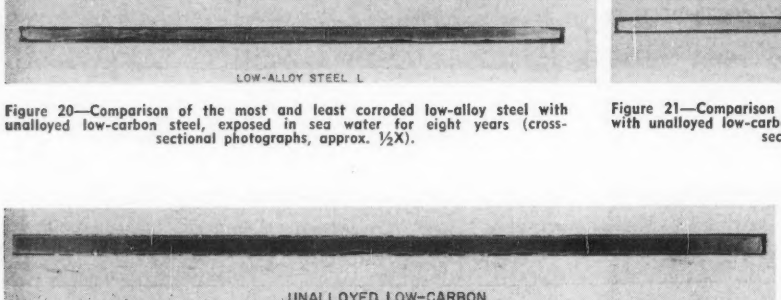


Figure 22—Comparison of the most and the least corroded low-alloy steel with unalloyed low-carbon steel, exposed in Gatun Lake fresh water for eight years (cross-sectional photographs, approx. 2/3X).

mersed in Gatun Lake fresh water. Pitting attack was slightly less, and weight loss results were more than 50 percent less than for unalloyed low-carbon steel. However, in sea water, even though the severity of pitting was approximately the same on the three steels the frequency of pitting on the chromium steels was much greater. The weight loss of 3 percent and 5 percent chromium steels exceeded that of low-carbon unalloyed steel by 63 percent and 30 percent, respectively. Exposed to the action of mean tide, the weight loss of the chromium steels was the same as the mild steel, but the pitting attack in this environment was the worst of any of the steels tested and was over double that of the mild steel in severity, and immeasurably higher in frequency.

7. Proprietary low-alloy steels were not, in general, more resistant to underwater corrosion than the mild unalloyed carbon steel, and for certain conditions of exposure the formulated steels were considerably less resistant. The practice of using these steels for underwater structures on the basis of their atmospheric corrosion resistance is definitely uneconomical.

8. Short-term tests would have given entirely erroneous conclusions regarding corrosion resistance for many of the steels included in the investigation. Since the corrosion-time curves were not linear and of a different shape for each different environmental media, the true corrosion rates could not have been established with fewer periods or less exposure time.

9. The conclusion of other investigators that low-alloy additions were usually of no consequence in increasing the corrosion resistance of steels immersed in sea water was borne out in most instances by these tests. The harmful effects of some alloys, developing after long periods of exposure in these environments, were unexpected and should be considered before the selection of structural steels for use in tropical waters.

Acknowledgments

Special acknowledgment is made of the contributions of H. W. Mundt who helped plan much of the experimental work and who, until his death in 1953, was Scientist-in-Charge of the Canal Zone Corrosion Laboratory. Also, the contributions of L. J. Thompson are gratefully acknowledged. The support of The Panama Canal Company and the Engineer Research and Development Laboratories, Fort Belvoir, Va., has been indispensable in the course of this work. The interest of E. M. Browder and George Howard of these organizations respectively has been particularly helpful.

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Any discussion of this article not published above will appear in the June, 1960 issue.

Influence of Cold Work on

Hydrogen Behavior in Steel*

By R. M. HUDSON, K. J. RIEDY and G. L. STRAGAND

Introduction

WHEN HYDROGEN behavior in steel during and after immersion in acid was investigated, the hydrogen content in steel increased linearly with the square root of immersion time in acid until a saturation concentration was attained, after which the concentration did not increase with further time.[1] Lowering the pH slightly increased the saturation value and greatly increased the absorption rate. The saturation value was small for hot rolled steel but increased markedly with cold work. Annealing of cold worked steel decreased the saturation value.

Attention has been given to the pronounced role of the interaction of nitrogen and of hydrogen in iron and steel with imperfections.[2] Various types of imperfections occur in an iron crystal lattice: precipitates or inclusions, grain boundaries, voids that may or may not be associated with inclusions, vacancies and dislocations either individual or lined up to form small-angle boundaries. Interstitial atoms such as nitrogen or hydrogen not only occupy normal interstitial sites but also have a strong tendency to accumulate at grain boundaries, dislocations and other points of imperfection. The motion of dislocations is plastic deformation; when this motion is hindered, the metal is strengthened. Thus, the interaction of interstitial atoms, which hinders this motion, is important in the mechanical behavior of metals. The marked effect of cold work on hydrogen solubility results from the dislocations generated by cold work.[2] At low temperature, where hydrogen clusters in the dislocations, the solubility is much greater in cold-worked specimens than in annealed specimens; at higher temperatures this difference is not as great.

The effect of cold work on density and hydrogen-occulusive capacity for high-purity iron, ingot iron, and SAE 1020 steel has been determined by heating specimens in hydrogen gas.[3] Neither a density change nor any noteworthy change in hydrogen occlusion occurred with cold reduction or annealing of high-purity iron. Cold reduction decreased the density of ingot iron but did not change hydrogen occlusion in the metal. Cold reduction decreased the density of SAE 1020 steel; the hydrogen occlusion at 250 C was increased about 100 times by cold reduction.

In a study of the influence of cold deformation on the rate of passage of electrolytically charged hydrogen through low-carbon sheet-steel diaphragms, this passage rate increased with increasing cold work up to 10 to 20 percent reduction and decreased rapidly with further deformation.[4]

Recent laboratory work concerning the influence of oxidizing and other environments on the rate of desorption of hydrogen from steel precharged with hydrogen established that environmental conditions had considerable effect on the rate of hydrogen desorption from steel.[5] Oxidizing agents actually hinder the desorption process whereas immersion of steel in water at 38 C resulted in the highest hydrogen desorption rate from steel for any of the treatments studied. For environments containing appreciable moisture, a second-order rate law applies; for environments containing extremely low amounts of moisture, a first-order rate law applies.

The time required for hydrogen desorption from steel in a dry environment was significantly longer than the time required when steel was immersed in water. A sequence of surface reactions has been suggested that may account for the catalytic effect of water on the evolution of hydrogen from steel.[6] The fact that such large differences in hydrogen-desorption rate exist shows that surface reactions can be rate determining. Therefore, in work on the influence of steel processing and other factors on hydrogen desorption from steel, environment control during all experiments will be essential.

The present study, concerning the influence of cold work on hydrogen-absorption rate into, solubility level in and desorption rate from steel was undertaken because of the practical importance of hydrogen in steel. Embrittlement of steel by hydrogen can be manifested by microscopic cracks (flaking), hairline cracks or blister formation or simply by a condition of low ductility after the steel is exposed to hydrogen charging. Hydrogen that enters steel by the iron-steam reaction during vitreous enameling is a principal cause of delayed defects encountered in enamelware.[7]

Discussion of Experiment

Materials

Samples of a commercial mild steel (Steel C) were obtained in the as-cold-reduced, as-continuously-annealed and as-box-annealed condition from the same original coil. The box-annealing operation involved a 4-hour hold period at 650 C; the continuous-annealing operation involved a hold period of less than a minute at this temperature. Thickness of this material was 0.010 inch. Speci-

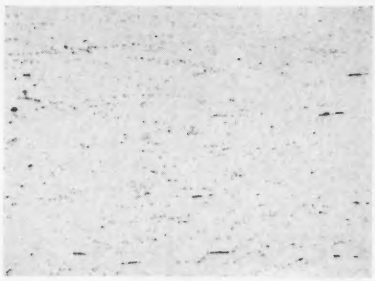


Figure 1—Cross section of Steel D (28.3 percent reduction) showing carbide structure. 500X, Picrol etch.



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Abstract

Discusses an investigation which shows that cold work markedly affects hydrogen's entry, solubility and desorption rate in steel. Also clarifies effect of processing variables on hydrogen behavior in steel. Includes discussion of materials, apparatus, environmental conditions and procedures used in experiments. 3.7.3

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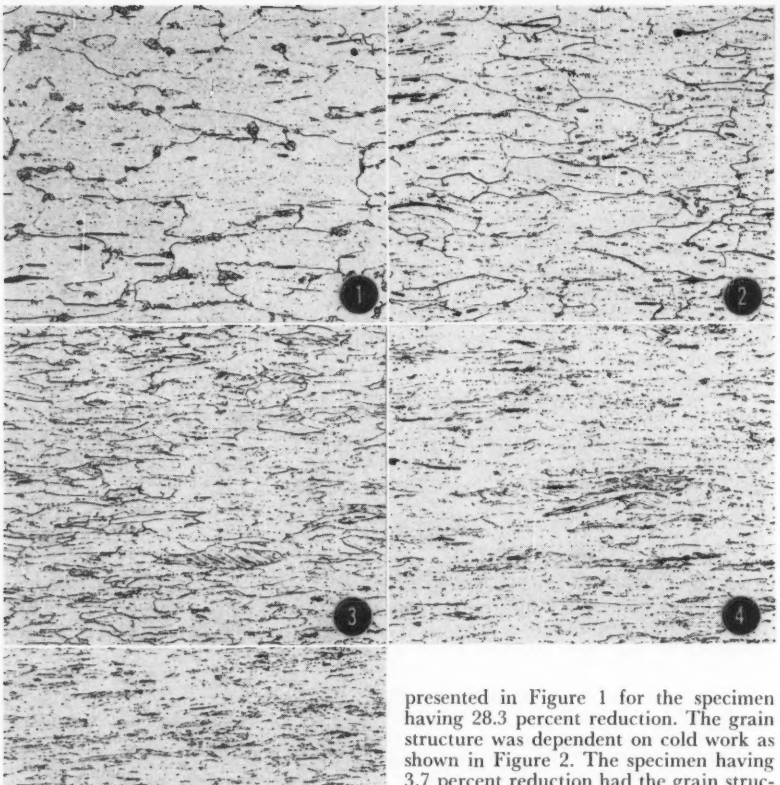


Figure 2—Grain structure of cold reduced specimens as a function of reduction percentage. 250X, Nital etch. Section 1: 3.7 percent. Section 2: 28.3 percent. Section 3: 47.3 percent. Section 4: 67.6 percent. Section 5: 78.2 percent.

mens were sheared to 2 by ½-inch coupons.

In the detailed study of the effect of cold work on hydrogen behavior in steel, low-carbon sheet-steel specimens were prepared in the laboratory representing reductions to 78.2 percent. From an original hot-rolled slab of Steel D of thickness 0.086 inch, a set of specimens were rolled at about 700 C to various thicknesses down to 0.0189 inch. All hot-rolled pieces were then given a light sandblasting treatment to remove scale and heat-treated for 1 hour at 650 C in a dry atmosphere containing 5 percent hydrogen and 95 percent nitrogen. All pieces were cold-reduced to a final thickness of about 0.02 inch. Specimens were sheared to 2 by ⅜-inch coupons.

Metallographic examination was made of this group of cold reduced samples to obtain information on carbide size, shape, distribution and grain structure. The carbides were small, spheroidal, intermediate in frequency and stringered for all samples. A photomicrograph showing carbides typical of those in all samples is presented in Figure 1 for the specimen having 28.3 percent reduction. The grain structure was dependent on cold work as shown in Figure 2. The specimen having 3.7 percent reduction had the grain structure of hot-rolled steel with only slight distortion in the direction of rolling. The specimen having 28.3 percent reduction displayed significant elongation of grain boundaries. The specimen having 47.3 percent reduction had some vestige of the hot-rolled grain structure although further elongation of grain boundaries was achieved. The specimens having 67.6 and 78.2 percent reduction displayed a completely fibrous structure with essentially no grain boundaries.

Chemical analyses for the steels used in this investigation are given in Table 1. Individual specimens were vapor-degreased with trichloroethylene and stored in a desiccator until ready for use. Nitrogen gas containing less than 0.01 percent oxygen was used for deoxygenating water and for experiments in which desorption of hydrogen from steel held in a dried nitrogen atmosphere was studied. Nitrogen gas was dried by successive passage through columns packed with solid desiccants and finally through a liquid nitrogen trap. Reagent grade sulfuric acid was used to prepare the acid solutions used for charging steel with hydrogen.

Apparatus and Procedure

Specimens to be charged with hydrogen were immersed in 2N sulfuric acid solutions at 38 C for various times. Hydrogen contents in charged-steel specimens and in specimens given treatments subsequent to charging were determined by the warm-extraction method.[8] Specimens were placed in specially constructed borosilicate-glass extractors and heated for 16 hours at 160 C under mercury.

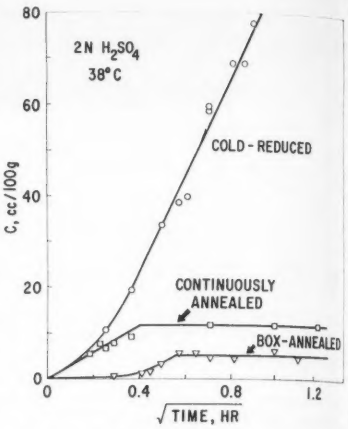


Figure 3—Hydrogen-absorption rate curves for Steel C.

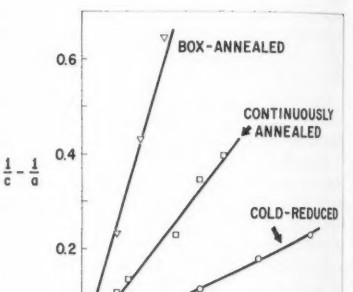


Figure 4—Hydrogen removal from Steel C in water at 38 C.

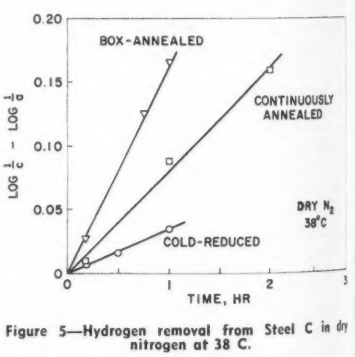


Figure 5—Hydrogen removal from Steel C in dry nitrogen at 38 C.

TABLE 1—Chemical Composition of Steels

Steel	COMPOSITION, PERCENT									
	C	Mn	P	S	Si	Cu	Ni	Cr	N	As
C.....	0.07	0.33	0.011	0.028	0.010	0.07	0.05	0.04	0.005	0.006
D.....	0.14	0.48	0.009	0.030	0.013	0.02	0.01	0.02	0.010	0.008

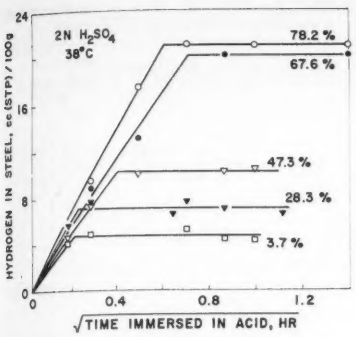


Figure 6—Hydrogen-absorption rate curves as a function of cold work.

Hydrogen extracted from the steel by this method was collected over mercury and measured volumetrically. Hydrogen removed by warm extraction and at temperatures to 160 C was considered to be primarily from the iron lattice and not from voids within the steel. This article is concerned, therefore, only with lattice-contained hydrogen.

Environmental Conditions

The usual procedure was to immerse steel specimens in 2N sulfuric acid at 38 C for a certain time. Comparisons of hydrogen desorption rates between different specimens in a group were made in most instances on the basis of similar weight losses during the pickling. Thus, though different initial hydrogen amounts were introduced into the different specimens during pickling, steel surface etching by pickling was probably similar. After charging, specimens were removed from the acid solution and thoroughly rinsed with running tap water for 15 seconds. Specimens were then given a final rinse with distilled water for 5 seconds before holding them for prescribed times under the various environmental conditions studied.

The rate of hydrogen desorption from steel specimens was studied in water and in dried nitrogen at 38 C. When the specimens were to be held in dried nitrogen, an additional drying treatment was used that consisted of rinsing the specimens with acetone followed by air drying. Not more than a minute was required for the operations conducted after acid charging and before isolation in controlled environments.

Following storage under deoxygenated water or dried nitrogen, all specimens were rinsed finally with distilled water for 5 seconds before determination of residual hydrogen content in the steel by the warm-extraction technique.

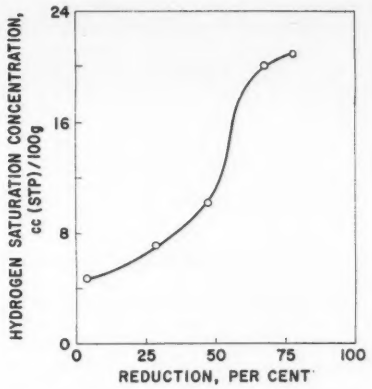


Figure 7—Change in hydrogen solubility with cold work.

Results and Discussion

Influence of Steel-Processing Condition on Hydrogen Absorption During Pickling

For Steel C in 2N sulfuric acid at 38 C, curves of hydrogen-charging rate were determined in the cold-reduced, continuously annealed and box-annealed conditions. These absorption rate curves are shown in Figure 3, and information derived from these curves is presented in Table 2. In agreement with other findings,[1,5,8] the hydrogen concentration increased linearly with the square root of the time the specimen was immersed in acid until a saturation concentration was attained. The cold-reduced specimen of Steel C, prepared from a hot-rolled strip of about 0.080-inch thickness (87.5 percent reduction), was not charged to a saturation level because, after longer immersion times in acid than those shown in Figure 3, the specimens were severely corroded and blistered by the acid and the measured hydrogen concentrations became erratic.

The actual saturation concentration was greatest for the cold-reduced steel and least for the box-annealed steel, the continuously annealed steel (which had a shorter heat-treatment than the box-annealed steel) having an intermediate saturation level. Increased time and temperature of heat-treatment caused a lowering in hydrogen-saturation concentrations reached by steel during pickling, according to other investigations.[1]

Influence of Steel-Processing Condition on Rate of Hydrogen Desorption

The desorption of hydrogen from Steel C in the cold-reduced, continuously annealed and box-annealed conditions during specimen immersion in deoxygenated water followed a second-order rate law as shown in Figure 4 where "c" is the residual hydrogen in the specimen at time "t," and "a" is the amount of hydrogen in the specimen after charging and prior to the rate-of-desorption study. Weight loss during pickling for the specimens used to obtain data shown in Figure 4 was 0.22, 0.23 and 0.21 percent, respectively, for the cold-reduced, continuously annealed and box-annealed specimens. The desorption of hydrogen from similar Steel C specimens during storage in dried nitrogen gas at 38 C followed a first-order rate law as shown in Figure 5 (base 10 logarithms were used). Rate constants derived from Figures 4 and 5 are given in Table 3.

From the data in Tables 2 and 3 and from Figures 3, 4 and 5, the cold-reduced specimens absorbed hydrogen during pickling more readily and to a higher concentration level than the annealed materials and the rate of hydrogen desorption decreased in the following order: box-annealed, continuously annealed and cold-reduced. Rate of hydro-

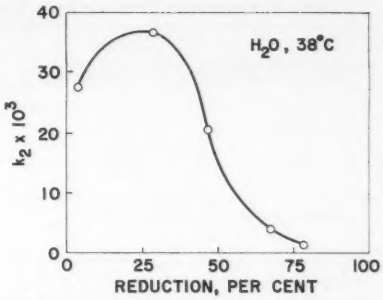


Figure 8—Change in removal rate in water at 38 C as a function of cold work.

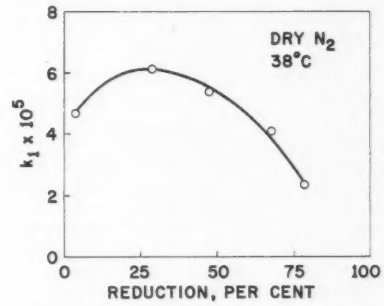


Figure 9—Change in removal rate in dry nitrogen at 38 C as a function of cold work.

TABLE 2—Hydrogen-Charging Rate for Steel C During Pickling as a Function of Processing Condition*

CONDITION	Time to Reach Saturation Level, min	Hydrogen Saturation Concentration, cc/100 g
Cold-reduced	>50	>78
Continuously annealed	10	12
Box-annealed	19	5

*Pickling was in 2N H_2SO_4 at 38 C.

TABLE 3—Rate of Hydrogen Desorption From Steel C as a Function of Processing Condition

CONDITION	Time Pickled in 2N H_2SO_4 at 38 C, min	Hydrogen Concentration After Pickling, cc/100 g	Weight Loss, %	DESORPTION RATE*	
				Water at 38 C, $k_2 \times 10^3$	Dry N_2 at 38 C, $k_1 \times 10^5$
Cold-reduced	8	19	0.22	6.4	2.2
Continuously annealed	15	12	0.23	18.2	5.2
Box-annealed	20	5	0.21	55.8	10.7

* Unit for k_2 is g/cc(STP)sec.
Unit for k_1 is reciprocal seconds.

TABLE 4—Hydrogen-Charging Rate During Pickling as a Function of Cold Work*

Specimen Thickness as Hot-Rolled, in.	Specimen Thickness as Cold Reduced, in.	Reduction, %	Time to Reach Saturation Level, min	Hydrogen Saturation Concentration cc/100 g
0.0189	0.0182	3.7	2.8	4.7
0.0265	0.0190	28.3	3.3	7.1
0.0385	0.0203	47.3	9.6	10.2
0.0599	0.0194	67.6	30.2	20.0
0.0849	0.0185	78.2	22.3	20.9

* Pickling was in 2N H_2SO_4 at 38 C.

gen desorption from steel specimens stored in dried nitrogen gas at 38 C was slow relative to corresponding rates in water at 38 C; a finding in agreement with earlier work.[5] Rate of hydrogen desorption from steel stored in dried nitrogen decreased in the following order: box-annealed, continuously annealed and cold-reduced.

Effect of Degree of Cold Work on Hydrogen Absorption During Pickling

Curves of hydrogen-absorption rate were obtained for specimens of Steel D having 3.7, 28.3, 47.3, 67.6 and 78.2 percent reductions (Figure 6). These curves were obtained for specimens immersed in 2N sulfuric acid at 38 C. Hydrogen saturation concentrations, given in Table 4 and shown in Figure 7 as a function of cold work, increased with increasing amount of cold work, in agreement with other findings.[1]

Effect of Degree of Cold Work on Rate of Hydrogen Desorption

For studies on the rate of hydrogen desorption, specimens of Steel D were given an initial hydrogen charging by immersion in 2N sulfuric acid at 38 C. Although the initial concentration of hydrogen in these specimens was not the same, as shown in Table 5, weight losses were between 2.1 and 2.5 percent. Etching of the steel surface during pickling therefore can be assumed to be similar for all lots of steel. Desorption rate was determined for steel immersed in water at 38 C and for steel stored in dried nitrogen at 38 C.

The desorption rate in water followed a second-order law; the rate in dried nitrogen followed a first-order law, agreeing with previous studies.[5] The rate constants derived from the usual plots $\left(\frac{1}{c}\right.$ vs t for water and $\ln \frac{1}{c}$ vs t for dried nitrogen) are given in Table 5 and are shown in Figures 8 and 9 as a function of cold work. For both environmental conditions studied, it was determined that with increasing cold work the desorption rate increased to a maximum from 20 to 30 percent cold reduction and that further increase in reduction resulted in a decrease in desorption rate.

Rate of passage of electrolytically charged hydrogen through low-carbon sheet-steel diaphragms has been found to increase with increasing cold work from 10 to 20 percent reduction and this rate decreased rapidly with further cold work.[4] Thus, for low-carbon steels, not only does severe cold work produce an increase in hydrogen solubility in the metal as introduced by acid pickling, but the rate at which hydrogen so introduced into the steel can be subsequently removed is less for severely cold-worked material. Persons concerned with the practical problem of embrittlement of steel as a result of acid pickling should exercise special care to minimize the introduction of hydrogen into cold-worked material.

TABLE 5—Rate of Hydrogen Desorption as a Function of Cold Work

Reduction, %	After 30-Minute Pickle in 2N H_2SO_4 at 38 C: Hydrogen Concentration cc/100 g	After 30-Minute Pickle in 2N H_2SO_4 at 38 C: Weight Loss, %	Desorption Rate*: Water at 38 C $k_2 \times 10^3$	Desorption Rate*: Dry N_2 at 38C $k_1 \times 10^3$
3.7	4.7	2.5	27.3	4.7
28.3	7.1	2.4	36.8	6.1
47.3	10.2	2.1	20.6	5.4
67.6	20.0	2.3	4.1	4.1
78.2	20.9	2.2	1.4	2.4

* Unit for k_2 is g/cc(STP)sec.
Unit for k_1 is reciprocal seconds.

Summary

This investigation has confirmed earlier results[5] showing the importance of steel surface reactions in the desorption of hydrogen from steel. The effect of increasing cold work in increasing the hydrogen solubility in steel following acid pickling was confirmed, as reported in the literature.[1,3] A maximum value of hydrogen desorption rate in water and in dry nitrogen at 38 C was observed at from 20 to 30 percent reduction. Severely cold-worked steel displayed a considerably lower desorption rate than less cold-worked steel or annealed materials, regardless of whether the environment was water or dried nitrogen.

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Corrosion and Weldability Studies On Chromium-Manganese Austenitic Stainless Steels*

By DONALD WARREN

Abstract

Corrosion and weldability studies have been made on Cr-Mn-Ni and Cr-Mn-N grades of austenitic stainless steel to determine their usefulness for chemical plant applications. These studies show that the corrosion resistance of Type 202 (18% Cr-5% Ni-8% Mn-0.10% max. C) stainless steel is comparable to that of Type 304 stainless steel in all but the most strongly oxidizing chemical environments. The weldability of the Cr-Mn-Ni and Cr-Mn-N steels is good and equivalent to that of Type 304 stainless steel. Includes history of chromium-manganese stainless steels. Tabular data included on tests conducted.

6.2.5

Introduction

AUSTENITIC STAINLESS steels are important materials of construction in the chemical industry because of their excellent corrosion resistance, formability and weldability. During the past decade, stainless steel producers have introduced several new lean-nickel grades of austenitic stainless steels, in which all or part of the nickel has been replaced with manganese and nitrogen.

Purpose of the investigation described in this article was to determine the suitability of the Cr-Mn-Ni and Cr-Mn-N austenitic stainless steels for chemical plant construction. The evaluation of these steels was based primarily on studies of their corrosion resistance and weldability.

History of Chromium-Manganese Stainless Steels

Recent technical publications and trade literature have described the lean-nickel or Cr-Mn-Ni steels as new stainless steels. Technically, these steels are not new since they were first introduced in the 1930's.[1,2] However, the Cr-Mn-Ni steels are new in terms of economic importance and commercial application.

The first stainless steels were of the straight 12-14 percent Cr variety and were ferritic (or body-centered-cubic) in structure. These were followed by the 18 percent Cr-8 percent Ni stainless steels which were austenitic (or face-centered-cubic) in structure. The addition of nickel markedly improved the corrosion resistance of the stainless alloys. However, the austenitic structure of the nickel-bearing stainless steels probably contributed as much to their widespread commercial application as did their corrosion resistance. The formability and weldability of austenitic stainless steel are outstanding compared to that of ferritic, straight-chromium stainless steel.

Unfortunately, there are only a limited number of elements which promote an austenitic structure when added to iron-chromium alloys. These austenite-forming elements are C, Ni, Mn, N, and Cu. The use of carbon is limited to small amounts(1) because of its deleterious effect on the corrosion resistance. Copper is not very effective as an austenite-former and contributes to hot-shortness if used in large amounts. Thus, only Ni, Mn and N are of real usefulness as austenite formers in stainless steel.

The first experiments using manganese to replace nickel in austenitic stainless steel were made in this country in 1930.[1] In the period between 1930 and 1942 extensive research was done on the Cr-Mn-Ni stainless steels both in this country and in Germany.[5-11] This work showed that without nickel, a 13 percent Cr stainless steel required at least 14 percent Mn to be austenitic. At chromium contents above 13 percent, nickel must be added with the manganese to produce an austenitic stainless steel. Additional investigations of the Cr-Mn-Ni steels showed that nitrogen was effective in promoting an austenitic structure.[12-14]

By 1943, a 15 percent Cr-1.5 percent Ni-12 percent Mn stainless steel was used in the German dairy and beer industry and in household appliance manufacture.[15] However, in this country there was little commercial application of the Cr-Mn-Ni steels before World War II, primarily because there was no real shortage of nickel. During World War II, a 17 percent Cr-4 percent Ni-4 percent Mn steel containing 0.10-0.15 percent N and 0.08-0.12 percent C was used in the American transportation industry in place of Type 301 stainless. The austenitic structure of this steel was relatively unstable, resulting in excessive work-hardening and frequent breakage during cold-forming operations. Later, it was shown that raising the manganese content to 6 percent or more would stabilize the austenite in the 17 percent Cr-4 percent Ni steels and lower their rate of work-hardening.

During the 1951 Korean emergency, the National Production Authority issued Directive M-80, limiting the use of nickel in most stainless steels for civilian consumption to 1 percent maximum. As a result of this directive, a stainless alloy containing 15 percent Cr-1 percent Ni-17 percent Mn with 0.10-0.20 percent N and 0.08-0.15 percent C was introduced by the steel producers.[16-19] This alloy, used widely in the transportation industry, was designated by the Budd Company as Trailer-Rail-Car steel (TRC steel).[17] Although the mechanical properties of this steel were comparable to those of Type 301 stainless, its corrosion resistance and hot workability were inferior.[16,19]

Although government limitations on the use of nickel in stainless steel were lifted after the Korean emergency, nickel remained in short supply because of government stockpiling and the expanding requirements of industry for nickel-bearing alloys.[20,21] Stainless steel producers renewed their production of a 17 percent Cr-4 percent Ni-6 percent Mn steel as a substitute for Type 301. In 1955, the American Iron and Steel Institute officially introduced two tentative grades of Cr-Mn-Ni stainless steel: Type 201, 17 percent Cr-4 percent Ni-6 percent Mn, and Type 202, 18 percent Cr-5 percent Ni-8 percent Mn.[22] The nominal chemical compositions of these steels are given in Table 1 with those for other common types of Cr-Mn-Ni steels. Several papers have reviewed the industrial applications of Types 201 and 202 stainless steel.[23-26] The corrosion resistance of the Cr-Mn-Ni steels also has been extensively studied.[3,27-29]

Recent research has clarified the structural relationships involved in melting and rolling of austenitic stainless steel.[30,31] From this research has come development of a nickel-less grade, Cr-Mn-N austenitic stainless steel.[30,32] This alloy has a nominal composition of 17 percent Cr-15 percent Mn-0.40 percent N with only residual amounts of nickel (less than 0.5 percent).

Previously, the use of nitrogen as an austenite-former in stainless steel was

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(1) 0.10 percent max. C for general corrosion resistance.[3] 0.03 percent max. C for resistance to intergranular corrosion in the as-welded condition.[4]

limited to 0.25 percent or less because of excessive gas evolution during solidification of the ingot. However, it was learned subsequently that solubility of nitrogen in austenitic stainless steel could be increased by maintaining a Cr content of 17 percent and increasing the Mn content to 15 percent (roughly, the solubility of $N = 1/100\ (\%Cr + \%Mn)$).[30]

Outline of Investigation

This investigation was concerned with the evaluation of the corrosion resistance and weldability of the lean-nickel grades of austenitic stainless steel.

Corrosion resistance of four grades of lean-nickel stainless steel was evaluated and compared with that of Type 430 and Type 304L stainless steel. The four grades of lean-nickel steel were Cr-Mn-N, TRC steel, Type 201 and Type 202.

The corrosive environments used in the evaluation can be divided into four classes: (a) environments typical of those for which du Pont uses Type 304 stainless steel (cold organic acids, hot and cold nitric acid solutions and environments which produce rusting or product contamination), (b) more severely corrosive environments in which Type 304 corrodes at a rate of 1-2 mils/mo. or in which Type 304 displays borderline passivity,[2] (c) pitting tests and (d) intergranular corrosison studies in which the standard nitric acid test was used to evaluate the influence of heat-treatment. Most of the corrosion studies were laboratory tests. However, two field exposures were made: one in a process dryer and the other in two nitric-adipic acid solutions.

Weldability of Cr-Mn-N and Type 202 stainless steel was evaluated in comparison to that of Type 304L stainless.[3] All three materials were evaluated for the metallic-arc and tungsten inert-arc welding processes according to standard qualification procedure for ferrous material as given in the ASME Boiler and Pressure Vessel Code, Section 9, Part A. The evaluation included radiographic examination, guided bend tests and tensile tests. Welded samples of Type 202, Type 202L and Type 304L stainless steel also were subjected to the standard nitric acid corrosion test.

Experimental Details

Materials

Materials used in this research included Type 430, Cr-Mn-N, TRC steel, Type 201, Type 202, Type 202L and Type 304L stainless steel. All materials were in the form of commercially annealed sheet or plate. Table 2 gives the chemical compositions of the steels used, along with the respective sheet or plate thickness. Check carbon analyses are included for those steels used in the intergranular corrosion studies.

Corrosion Tests

Sample Size and Preparation. Corrosion specimens were approximately 1-inch square except those used in the pitting tests which were 1-inch wide by 2 inches long. All specimens were given an 80-grit finish by belt-grinding before testing. Samples in the pitting test were evaluated in both the pickled and the as-ground conditions. The 80 C pickling solution consisted of 104 ml of 65% HNO_3, 17 ml of 37% HCl, 17 ml of 48% HF and 862 ml of H_2O.

Heat-Treatment of Samples. Except for the intergranular corrosion studies and one field exposure, all corrosion specimens were tested in the commercially annealed condition. Heat-treatment of specimens for the intergranular corrosion studies was done in a combustion type tube furnace; the specimen temperature was controlled to within ±10 F. An argon atmosphere was used for heat-treatments at temperatures above 1300 F to minimize scaling and decarburization.

Corrosive Environments. Table 3 lists the corrosive environments used for evaluating the corrosion resistance of the lean-nickel stainless steels. The environments designated as "mildly to moderately corrosive" are typical of those for which du Pont uses Type 304 stainless steel. The same is true for the two field exposure environments involving nitric-adipic acid solutions. The "more severely corrosive" environments were selected in order to evaluate the lean-nickel steels under more adverse conditions. The purpose of the pitting, rusting, and intergranular corrosion tests is self-evident.

Procedures for General Corrosion Tests.

General Procedure. All specimens were individually tested in flasks or glass cylinders containing a minimum 250 ml of the test solution. Fresh solution was used for each test period. Length and number of corrosion periods varied according to the nature and severity of the test. All tests were conducted for a minimum of 24 hours. Rusting tests and the field exposures were much longer in duration.

Cleaning of Samples. Specimens were cleaned after each test period with water and liquid soap. Loosely adherent cor-

[2] Borderline passivity as used in this paper refers to the following type of behavior: A group of duplicate specimens of the same steel and having the same surface treatment is exposed to a given corrosive environment. Some of the specimens remain passive and do not corrode. The remaining specimens become active and corrode at a measurable rate.

[3] The amount of available T.R.C. steel and Type 201 stainless steel was insufficient for a weldability study.

TABLE 1—Common Types of Chromium-Manganese Austenitic Stainless Steels and Their Nominal Chemical Compositions

Type of Steel	Descriptive Designation (Cr-Ni-Mn)	NOMINAL COMPOSITION, %				
		Cr	Ni	Mn	N	C
Cr-Mn-N	17-0-15	17.0	.....	14.5	0.40	0.10
TRC*	15-1-17	15.0 min.	1.0 max.	16.5	0.15	0.15 max.
	16-1-16					
201	17-4-6	16.0–18.0	3.5–5.5	5.5–7.5	0.25 max.	0.15 max.
202	18-5-8	17.0–19.0	4.0–6.0	7.5–10.0	0.25 max.	0.15 max.

* "Trailer-Rail-Car" steel is a commercial designation originated by the Budd Company

TABLE 2—Chemical Composition of Stainless Steel Heats Used for Corrosion and Weldability Tests

Type of Steel	Heat Number[1]	Sheet or Plate Thickness, in.	CHEMICAL COMPOSITION, %								
			C (Reported)[2]	C (Check)[3]	Cr	Ni	Mn	N	Si	P	S
430	GU8	0.125	0.06	.....	17.28	.....	0.34	.....	0.40	0.019	0.013
Cr-Mn-N	HK7	0.080	0.10	.....	17.76	0.24	15.50	0.41	0.36	0.030	0.007
Cr-Mn-N	HQ5	0.170	0.06	.....	18.07	0.52	15.20	0.51	0.10	0.022	0.008
TRC[4]	HD4	0.090	0.10	.....	15.05	1.07	16.50	0.16	0.56	0.030	0.016
TRC[4]	HO1	0.060	0.08	.....	15.26	1.03	16.80	.....	0.32	0.036	0.007
201	GZ7	0.035	0.11	.....	17.60	4.39	5.60	0.14	0.24	0.030	0.009
202	HO3	0.230	0.065	0.060	18.39	5.38	9.03	0.23	0.57	0.018	0.009
202	HN2	0.060	0.035	0.036	19.00	4.97	9.54	0.27	0.49	0.009	0.011
202L	HN3	0.060	0.030	0.030	18.59	4.91	9.61	0.28	0.61	0.008	0.015
202L	HP4	0.120	0.024	0.021	17.22	5.39	9.99	0.20	0.64	0.024	0.010
202L	HO4	0.280	0.025	0.028	17.18	5.05	9.65	0.20	0.64	.....	.....
304L	FL4A	0.135	0.026	.....	18.19	9.48	1.11	0.13	0.33	.....	.....
304L	FL3A	0.190	0.020	.....	18.21	9.77	0.76	0.13	0.47	.....	.....
304L	FL5A	0.165	0.016	.....	18.48	13.40	1.11	0.029	0.40	.....	.....
304L	FE5A	0.140	0.021	.....	20.00	10.50	1.23	0.032	0.46	.....	.....
304L	FK4A	0.265	0.020	.....	18.30	11.02	1.06	0.033	0.37	.....	.....

[1] All heats were commercially produced and were received in the annealed condition.
[2] Ladle or heat analyses as reported by the producer.
[3] Laboratory analyses based on three or more determinations with a Leco Carbon Conductrometric Determinator.
[4] "Trailer-Rail-Car" steel is a commercial designation originated by the Budd Company.

rosion product films were removed with a nonabrasive cleaning powder. In the 10 percent oxalic acid test, a room-temperature solution of 65 percent nitric acid was used to remove the oxalate corrosion product.

Activation of Samples. Activation of corrosion specimens before exposure in the test solution can be used to develop information about: (a) passivating power of the test solution, (b) effect of alloy composition on the development of passivity and (c) behavior to be expected from the alloy if passivity is destroyed in service.

Consequently, except for the two nitric acid tests,[4] all tests in the group of mildly-to-moderately corrosive environments were made with and without prior activation. The specimens were activated electrolytically before each test period by the cathodic liberation of hydrogen for 10 to 15 seconds. Activation consisted of applying a 2 to 3-volt emf across an electrolytic cell with the corrosion specimen as the cathode, the test solution as the electrolyte and a stainless steel wire as the anode.

Three tests in the group of more severely corrosive environments also were made using prior activation. For the boiling 25 percent phosphoric acid test, specimens were activated by immersion in a 65 C solution of 15 percent hydrochloric acid (by volume[5]) until hydrogen was evolved from most of the specimen surface.[33] Following activation, the samples were rinsed successively in two flasks of distilled water and in a flask of boiling 25 percent phosphoric acid before immersion in the phosphoric acid test solution. Weight losses for this activation method were less than 1 mg.

Recording of Corrosion Data. The progress of corrosion was checked by weight-loss measurements and by visual and microscopic observation made after each test period. All corrosion rates except those for the boiling 65 percent nitric acid test are reported as mils/mo. Results of the boiling 65% nitric acid tests are given as in./mo. (standard procedure for this test).

[4] Nitric acid is a strong oxidizing agent. Consequently, any activated specimen would soon be passivated by the nitric acid test solution.

TABLE 3—Corrosive Environments Used for Evaluation of Chromium-Manganese Austenitic Stainless Steels

Mildly to Moderately Corrosive Environments
1. Room Temperature Tests
a. 10% Formic Acid
b. 10% Oxalic Acid
c. 30% Acetic Acid
d. Glacial Acetic Acid
e. 1.5% Adipic Acid
f. 95% Sulfuric Acid
g. 90% Nitric Acid
2. 65% Nitric Acid at 60 C
3. Field Exposure: 45% Nitric-22% Adipic Acid Solution at 55 C
More Severely Corrosive Environments
1. 30% Nitric Acid—Boiling
2. 65% Nitric Acid—Boiling
3. 25% Phosphoric Acid—Boiling
4. Nitrating Acid (80% H_2SO_4—15% HNO_3—5% H_2O) at 80 C
5. 5% Acetic Acid at 80 C
6. 5% Lactic Acid at 80 C
7. 90% Formic Acid at 60 C
8. Field Exposure: 40% Nitric—20% Adipic Acid Solution at 95 C
Pitting Tests
1. Aerated 4% Sodium Chloride Solution at 85 C
2. 5% Ferric Chloride Solution at Room Temperature
Tests for Rust Resistance
1. Weatherometer Test: Alternate Water Spray-Drying Cycle at 40 C
2. Steam Sparging Unit: 100% Relative Humidity at 99-100 C
3. Field Exposure in a Process Dryer: Water Vapor Plus Traces of Sulfuric Acid at 160 C
Intergranular Corrosion Tests
1. Electrolytic Oxalic Acid Etching Test
2. Standard Boiling 65% Nitric Acid Test

Procedures for Intergranular Corrosion Tests.

Electrolytic Oxalic Acid Etching Test. Standard ASTM procedure[34] was followed in making the electrolytic oxalic acid etching test. The surface to be examined was prepared by grinding through No. 3/0 emery paper. The specimen then was electrolytically etched in a 10 percent oxalic acid solution for 1.5 minutes at a current density of 1 amp/sq cm. Finally, the etched surface was examined at a magnification of 250-500X.

Standard Nitric Acid Test. The standard boiling 65 percent nitric acid test was made according to ASTM procedure.[35] Specimens were tested in individual flasks for five 48-hour periods.

Weldability Studies

Materials. One heat of Cr-Mn-N and two heats each of Type 202 and Type 202L stainless steel were available in sufficient quantity for making weldability studies. A Type 304L heat was included as a standard of comparison.

[5] 5.4 percent by weight.

Procedure. All the above materials were evaluated for metallic-arc and tungsten inert-arc welding processes according to standard qualification procedure for ferrous material as given in ASME Boiler and Pressure Vessel Code, Section 9, Part A. Evaluation included radiographic examination, guided bend tests and tensile tests.

Test plates were made up by butt welding in the longitudinal direction two strips of sheet or plate, 5 inches wide by 14 inches long. Single V-type joints were used for heavier gage material and square butt joints for the lighter. Joint edges were prepared by grinding. Type 308 coated electrodes or Type 308 filler wire was used for welding both of the Type 202 heats, one Type 202L heat and the Cr-Mn-N heat. Type 308L coated electrodes or Type 308L filler wire was used for welding the other Type 202L heat and the one Type 304L heat. Data on the welding conditions for each test plate are given in Table 4. Each test plate provided two face bend specimens, two root bend specimens and two reduced-section tensile specimens.

Standard Nitric Acid Corrosion Testing of Welded Samples. The nitric acid corrosion resistance of welded and welded-and-annealed samples of two heats each of Type 202 and Type 202L was evaluated and compared with that of Type 304L stainless steel. (The Cr-Mn-N heat was not included in the nitric acid evaluation).

Test plates were made up by laying longitudinal bead-on-plate welds down the center of 2 inches wide by 6 inches long strips. Metallic-arc and tungsten-inert-arc welding processes were used. The welding electrodes were the same as those used in the weldability studies; however, no filler metal was used for any of the tungsten inert-arc welds. All welds were made at the highest heat inputs consistent with good welding practice to produce maximum carbide precipitation in the heat-affected zones.

Individual specimens about 1-inch square were cut from the center of each welded test plate. These were ground

TABLE 4—Data on Welding Conditions for Cr-Mn-N, Types 202 and 202L Stainless Steel

Type of Steel	Heat Number	Sheet or Plate Thickness, In.	Welding Process	Type of Joint	Type of Welding Electrode or Filler Wire	Shielding Gas and Flow Rate	Current, Amp.	Voltage	Number of Passes
Cr-Mn-N....	HQ5	0.170	Metallic-arc	Single Vee	5/32 in. Type 308	None	120	22	3
			Tungsten Inert-arc	Single Vee	3/32 in. Type 308	Argon, 15 c.f.h.	100	11	3
202.....	HO3	0.230	Metallic-arc	Single Vee	5/32 in. Type 308	None	125	22	3
			Tungsten Inert-arc	Single Vee	1/8 in. Type 308	Argon, 15 c.f.h.	140	13	3
202.....	HN2	0.060	Metallic-arc	Square Butt	3/32 in. Type 308	None	60	20	2
			Tungsten Inert-arc	Square Butt	1/16 in. Type 308	Argon, 10 c.f.h.	90	10	1
202L.....	HO4	0.280	Metallic-arc	Single Vee	5/32 in. Type 308	None	125	22	3
			Tungsten Inert-arc	Single Vee	1/8 in. Type 308	Argon, 15 c.f.h.	125	12	3
202L.....	HN3	0.060	Metallic-arc	Square Butt	3/32 in. Type 308L	None	60	20	2
			Tungsten Inert-arc	Square Butt	3/32 in. Type 308L	Argon, 12 c.f.h.	90	10	1
304L.....	FL3A	0.190	Metallic-arc	Single Vee	5/32 in. Type 308L	None	130	20	2
			Tungsten Inert-arc	Single Vee	1/8 in. Type 308L	Argon, 15 c.f.h.	120	12	3

TABLE 5—Corrosion Data for Tests in 10% Formic Acid at Room Temperature

Type of Steel	Heat Number	CORROSION RATE, MIL/MO		
		Activated Tests[1]		Non-Activated Tests[2]
		First 24-Hr. Period	Av. of Next Four 24-Hr. Periods	240-Hr. Period (After Regrind)
430 (17% Cr)	GU8	22	0	8
Cr-Mn-N	HK7	0.7	0.1	0
TRC	HD4	0.5	0.1	0.1
201	GZ7	0.7	0.1	0
304L	FL4A	1.1	0.2	0.5

[1] Samples were electrolytically activated in test solution prior to each test period. Samples were exposed for five 24-hour periods using fresh solution for each test period.
[2] Samples were reground and then exposed in fresh solution for one 240-hour period without prior activation.

TABLE 6—Corrosion Data for Tests in 10% Oxalic Acid at Room Temperature

Type of Steel	Heat Number	CORROSION RATE, MIL/MO		
		Activated Tests[1]		Non-Activated Test[2]
		Periods of Active Corrosion[3]	Average Rate[4]	240-hr Period (After Regrind)
430 (17% Cr)	GU8	Five	3.3	0
Cr-Mn-N	HK7	Five	0.7	0
TRC	HD4	Three	0.7	0
201	GZ7	One	0.7	0
304L	FL4A	Five	2.9	0
304L	FK4A	Five	2.2	0

[1] Samples were electrolytically activated in test solution prior to each test period. Samples were exposed for five 24-hour periods using fresh solution for each test period. A room temperature solution of 65% nitric acid was used to remove the corrosion product from FL4A and FK4A at the end of each test period. (There were no non-adherent corrosion products on specimens of the other four steels).
[2] Samples were reground and then exposed in fresh solution for one 240-hour period without prior activation.
[3] Samples corroded actively only for the number of periods indicated, i.e., GZ7 and HD4 became passive at the end of the first and third periods, respectively.
[4] Average 24-hour period rate based on those periods in which the sample was actively corroding.

TABLE 7—Corrosion Data for Tests in 30% Acetic Acid at Room Temperature

Type of Steel	Heat Number	CORROSION RATE, MIL/MO		
		Activated Tests[1]		Non-Activated Test[2]
		First 24-Hour Period	Average of Next Four 24-Hour Periods	240-Hour Period (After Regrind)
430 (17% Cr)	GU8	2.3	0	0
Cr-Mn-N	HK7	0.1	0	0
TRC	HD4	0.1	0	0
201	GZ7	0.1	0	0
304L	FL4A	0.1	0	0

[1] Samples were electrolytically activated in test solution prior to each test period. Samples were exposed for five 24-hour periods using fresh solution for each test period.
[2] Samples were reground and then exposed in fresh solution for one 240-hour period without prior activation.

TABLE 8—Corrosion Data for Tests in Glacial Acetic Acid at Room Temperature

Type of Steel	Heat Number	CORROSION RATE, MIL/MO FOR ACTIVATED TESTS[1]	
		First 24-Hour Period	Average of Next Four 24-Hour Periods
430 (17% Cr)	GU8	0.1	0
Cr-Mn-N	HK7	0.1	0
TRC	HD4	0.1	0
201	GZ7	0.1	0
304L	FL4A	0.1	0

[1] Samples were electrolytically activated in a 30% acetic acid solution at room temperature prior to each test period. (The low electrical conductivity of glacial acetic acid prevented electrolytic activation in the test solution.) Samples were exposed for five 24-hour periods using fresh solution for each test period.

smooth and flask-tested in boiling 65 percent nitric acid for five 48-hour periods. Similar specimens from three of the welded test plates were annealed 30 minutes at 1950 F, WQ and then nitric acid tested.

Discussion of Results

Corrosion Studies

Mildly to Moderately Corrosive Environments. The corrosion data for tests made in nine different environments of a mildly-to-moderately corrosive nature are reported in Tables 5-12. Five types of steel were included in all tests except the field exposure: Type 430 (17 percent Cr), Cr-Mn-N, TRC steel, Type 201, and Type 304L stainless steel. Results of each test are discussed individually below.

Formic Acid. Only the Type 430 stainless steel corroded at an excessive rate in 10 percent formic acid at room temperature (Table 5). All three of the lean-nickel grades (Cr-Mn-N ,TRC, and Type 201) were equivalent to Type 304(6) in their corrosion resistance. Corrosion rates for these austenitic stainless steels were all less than 0.5 mils/mo for test periods of 120 hours or longer whether or not prior activation was used.

Type 430 stainless steel underwent severe corrosion even without prior activation. However, Type 430 tended to become passive upon exposure to air following a period of severe corrosion.

Oxalic Acid. In the nonactivated or passive condition, all five types of steel were completely resistant to corrosion in 10 percent oxalic acid at room temperature (Table 6). However, in activated tests, the three lean-nickel steel grades were superior to Type 430 and Type 304 stainless steel. Corrosion rates for 24-hour activated tests on Type 430 and 304 stainless were four to five times those for lean-nickel steels. The lean-nickel steels showed strong self-passivating tendencies in the activated tests; i.e., the samples stopped corroding after the first few hours of each 24-hour test. These tendencies toward self-passivation are probably due to the high manganese content (6 to 17 percent) of the lean-nickel steels.

Acetic Acid. The three lean-nickel steels and Type 304 stainless steel showed excellent resistance to corrosion (rates of 0 to 0.1 mil/mo.) in both activated and nonactivated tests when exposed to 30 percent acetic acid at room temperature (Table 7). Although Type 430 stainless steel was completely resistant to attack in the nonactivated test, it corroded severely when activated before testing. Air exposure following a period of severe corrosion passivated the Type 430 stainless steel.

Glacial Acetic Acid. All five types of stainless steel were, for practical purposes, completely resistant to attack in glacial acetic acid at room temperature (Table 8).

Adipic Acid. With the exception of Cr-Mn-N steel, none of the steels in either the activated or nonactivated tests was attacked by 1.5 percent adipic acid at room temperature (Table 9). The Cr-Mn-N stainless steel was not attacked in the nonactivated test but underwent appreciable corrosion in the activated tests (0.40 mil/mo).

Sulfuric Acid. In nonactivated tests, all five steels showed good resistance to corrosion in 95 percent sulfuric acid at room temperature (Table 10). Type 304 stainless with no measurable corrosion rate was slightly superior to Type 430 and the three lean-nickel steels; corrosion rates for the latter four steels were 0.10 to 0.15 mil/mo.

In the activated 24-hour tests, all five steels corroded at high rates (1 to 4 mils/mo.). The activated 120-hour rate

(6) Whenever resistance to general corrosion is being discussed, the Type 304L heat of stainless steel will be considered as representative of the Type 304 (18% Cr-8% Ni) class of stainless steels. However, whenever intergranular corrosion resistance is discussed, the Type 304L (0.03% C max) heat will be distinguished from the regular carbon grade of Type 304 (0.08% C max). This same policy also applies in the use of the terms Type 202 and Type 202L.

TABLE 9—Corrosion Data for Tests in 1.5% Adipic Acid (Saturated Water Solution) at Room Temperature

Type of Steel	Heat Number	CORROSION RATE, MIL/MO. Activated Tests[1] Average of Five 24-Hour Periods	Non-Activated Test[2] 240-Hour Period (After Regrind)
430 (17% Cr)	GU8	0	0
Cr-Mn-N	HK7	0.40	0
TRC	HD4	0	0
201	GZ7	0	0
304L	FL4A	0	0

[1] Samples were electrolytically activated in test solution prior to each test period. Samples were exposed for five 24-hour periods using fresh solution for each test period.
[2] Samples were reground and then exposed in fresh solution for one 240-hour period without prior activation.

TABLE 10—Corrosion Data for Tests in 95% Sulfuric Acid at Room Temperature

Type of Steel	Heat Number	CORROSION RATE, MIL/MO Activated Tests[1] Average of Ten 24-Hour Periods	Activated Tests[1] 120-Hour Period	Non-Activated Test[2] 240-Hour Period (After Regrind)
430 (17% Cr)	GU8	3.1	0.3	0.1
Cr-Mn-N	HK7	4.1	0.2	0.1
TRC	HD4	3.8	0.4	0.15
201	GZ7	1.9	0.3	0.15
304L	FL4A	3.1	1.2	0

[1] Samples were electrolytically activated in test solution prior to each test period. Samples were exposed for ten 24-hour periods and then for one 120-hour period using fresh solution for each test period.
[2] Samples were reground and then exposed in fresh solution for one 240-hour period without prior activation.

TABLE 11—Corrosion Data for Tests in 90% Nitric Acid at Room Temperature and in 65% Nitric Acid at 60 C.

Type of Steel	Heat Number	CORROSION RATE, MIL/MO. 90% Nitric Acid, Room Temp.[1] Average of Ten 24-Hour Periods	65% Nitric Acid, 60 C[1] Average of Five 24-Hour Periods
430 (17% Cr)	GU8	0.2	0
Cr-Mn-N	HK7	0.3	0.2
TRC	HD4	0.3	0.4
201	GZ7	0.2	0
304L	FL4A	0.1	0

[1] Samples were exposed for the indicated number of 24-hour periods using fresh solution for each test period.

TABLE 12—Corrosion Data for Field Exposures in Nitric-Adipic Acid Solutions

Type of Steel	Condition	CORROSION RATE, MIL/MO, FOR THREE-MONTH EXPOSURE 40%Nitric-20% Adipic Acid Solution at 95 C	45% Nitric-22% Adipic Acid Solution at 55 C
202L[1]	Commercially Annealed	0.56	Nil[3]
	Sensitized 1 hour at 1250 F, WQ	0.62	Nil[3]
	Tungsten Inert-arc Welded	0.50	Nil[3]
304[2]	Commercially Annealed	0.18-0.24	Nil

[1] Heat HN3.
[2] Reported corrosion data were based on several heats of Type 304 stainless steel.
[3] Corrosion rates, although measureable, were all less than 0.03 mil/mo.

for each steel was lower than the activated 24-hour rate; this behavior indicated a tendency toward self-passivation with continued exposure in the test solution.

Ninety Percent Nitric Acid. All of the steels showed good resistance to attack (24-hour corrosion rates of 0.1 to 0.3 mil/mo) in 90 percent nitric acid at room temperature (Table 11).

Sixty-five Percent Nitric Acid. Type 430, Type 201 and Type 304 stainless were not measurably attacked in a 120-hour test in 65 percent nitric acid at 60C (0 mils/mo). Corrosion rates for Cr-Mn-N and TRC steel were low (0.2 to 0.4 mil/mo) (Table 11).

Field Exposure in Nitric-Adipic Acid. Both Type 202L and Type 304L stainless steel showed a nil corrosion rate after a three-month field exposure to 45 percent nitric-22 percent adipic acid at 55 C (Table 12). Type 430, Cr-Mn-N and TRC steel were not tested in this environment.

Summary of Test Results in Mildly-to-Moderately Corrosive Environments. These test results showed that the corrosion resistance of a 17 percent Cr-4 percent Ni, Type 201 stainless steel in mildly-to-moderately corrosive environments was comparable to that of Type 304 stainless steel. This conclusion agrees with the findings of other investigations[22, 29, 36] undertaken concurrently with the present work.

Corrosion resistance of Cr-Mn-N steel was also good in those environments tested. However, in the adipic acid solution and in the two nitric acid solutions, Cr-Mn-N steel corroded at a higher rate than Type 201 or Type 304 stainless steel.

TRC steel displayed corrosion resistance equivalent to Type 201 except in the two nitric acid environments. However, in general, TRC steel should not be considered comparable to Type 304 in mildly-to-moderately corrosive environments because of its low chromium content (15 percent). This latter viewpoint is shared by others.[16, 19]

Type 430 stainless steel corroded severely in three of the eight environments in which it was tested: 10 percent formic acid (activated or nonactivated), 30 percent acetic acid (activated), and 10 percent oxalic acid (activated). In the other five environments, Type 430 showed good resistance to corrosion.

The room-temperature tests in 10 percent oxalic acid demonstrated that manganese can contribute to the passivity of stainless steels in certain corrosive environments. All three of the lean-nickel steels containing 6 to 16 percent Mn were superior to Type 304 stainless steel in activated corrosion tests in the oxalic acid solution.

More Severely Corrosive Environments. Corrosion data for tests made in eight different environments of a more severely corrosive nature are reported in Tables 12 (40 percent nitric-20 percent adipic acid at 95 C) to 19. The stainless steels tested in these environments included Type 430, Cr-Mn-N, TRC steel, Type 202, Type 202L,(7) and Type 304L.(7)

Boiling 30 Percent Nitric Acid. The corrosion resistance of the individual steels in boiling 30 percent nitric acid depended largely upon their nickel content (Table 13). For example, the 240-hour corrosion rates for Type 304 (9.5 percent Ni), Type 201 (4.4 percent Ni), Cr-Mn-N (0.2 percent Ni) and Type 430 (0 percent Ni) were 0.22, 0.35, 0.71 and 0.56 mil/mo., respectively. Note that the corrosion rate for Type 201 is only 60 percent greater than that for Type 304 stainless steel even though it contains less than half as much nickel as the Type 304 heat. Previous work[29, 31] has shown the beneficial effect of nickel on the corrosion resistance of Cr-Mn-Ni stainless steel in strongly oxidizing environments. The first 4 percent of nickel added to Cr-Mn stainless steel produces the greatest improvement in resistance to corrosion in boiling nitric acid solutions; increasing the nickel to 6 to 8 percent provides some additional improvement.[29]

Boiling 65 Percent Nitric Acid. The corrosion resistance of the *annealed* lean-nickel stainless steels in boiling 65 percent nitric acid was governed mainly by their nickel and manganese contents (Table 14). The six heats of Type 201, 202 and 202L stainless containing 17.5 to 19.00 percent Cr and 4.4 to 5.4 percent Ni had nitric acid corrosion rates (1.3 to 1.8 mil/mo) that were approximately double those (0.8 mil/mo) for Type 304L (18 percent Cr-9.5 percent Ni). Corrosion rates for Cr-Mn-N (17.5 to 18.0 percent Cr and 0.2 to 0.5 percent Ni) were 7 to 8 times those for Type 304L. The T.R.C. steel (15 percent Cr and 1 percent Ni) had a corrosion rate slightly higher than that for Cr-Mn-N steel because of its lower chromium content.

(7) See Footnote 6.

TABLE 13—Corrosion Data for Tests in Boiling 30% Nitric Acid

Type of Steel	Heat Number	CORROSION RATE, MIL/MO.[1] Av. of Ten 24-Hr. Periods	240-Hr. Period (After Regrind)
430 (17% Cr)	GU8	0.59	0.56
Cr-Mn-N	HK7	0.62	0.71
TRC	HD4	....[2]	....[2]
201	GZ7	0.33	0.35
304L	FL4A	0.21	0.22

[1] Samples were exposed for ten 24-hr. periods, reground, and then exposed for one 240-hr period using fresh solution for each test period.
[2] Corrosion rates for HD4 are not included, because the sample underwent severe intergranular attack resulting in corrosion rates which increased with each test period. A second sample of HD4 which was laboratory reannealed for 30 min. at 1950 F, WQ, behaved in a similar fashion.

TABLE 14—Corrosion Data for Tests in Boiling 65% Nitric Acid

Type of Steel	Heat Number	CORROSION RATE, IN./MO. FOR STANDARD NITRIC ACID TEST[1] Annealed[2]	1 Hr, 1250 F, WQ	2 Hr, 1200 F, WQ	30 Min, 1950 F, WQ	30 Min, 1950 F, WQ + 1 hr, 1250 F WQ
430 (17% Cr)	GU8	0.0023	.....	.....	.....	.....
Cr-Mn-N	HK7	0.0060	0.062	.....	.....	.....
Cr-Mn-N	HQ5	0.0055	0.068	.....	.....	.....
TRC	HD4	0.019[3]	0.174	.....	.....	.....
TRC	HO1	0.0078	0.071[5]	.....	.....	.....
201	GZ7	0.0018	0.055[5]	.....	.....	.....
202	HO3	0.0013	0.078	0.13	0.0013	0.070
202	HN2	0.0015	0.0045	0.024	0.0015	0.0037
202L	HN3	0.0017	0.0023	0.0022	0.0013	0.0018
202L	HP4	0.0018	0.0019	0.0024	0.0020	0.0026
202L	HO4	0.0016	0.0020	0.0027	.....	.....
304L	FL4A	0.00079	0.00095	.....	.....	.....

[1] Based on individual flask tests in boiling 65% nitric acid for five 48-hour periods.
[2] All specimens were in the commercially annealed condition except for HD4 which was laboratory reannealed for 30 min. at 1950 F, WQ. Sensitized specimen of HD4 was also laboratory reannealed prior to the 1-hour at 1250 F heat-treatment.
[3] Specimen underwent severe intergranular attack and grain dropping despite the laboratory reannealing treatment. Commercially annealed sample behaved similarly.
[4] Corrosion rate based on two periods only.
[5] Corrosion rate based on three periods only.

TABLE 15—Corrosion Data for Tests in Boiling 25% Phosphoric Acid

Type of Steel	Heat Number	CORROSION RATE, MIL/MO. Activated Tests[1] 1st Period, 24 Hr.	2nd Period, 120 Hr.	3rd Period, 24 Hr.	Non-Activated Test[2] 120-Hr. Period
430 (17% Cr).....	GU8	7.5	1.8	0.2	0.2
Cr-Mn-N.....	HK7	0.9	0.2	0.5	....
Cr-Mn-N.....	HQ5	1.0	0.2	0.2	....
TRC.....	HD4	1.1	0.3	0.3	....
TRC.....	HO1	0.9	0.2	0.5	....
201.....	GZ7	0.3	0.1	0.2	....
202.....	HO3	0.4	0.2	0.2	....
202.....	HN2	0.2	0.1	0.1	....
202L.....	HN3	0.3	0.2	0.2	....
202L.....	HP4	0.3	0.2	0.1	....
202L.....	HO4	0.4	0.2	0.2	....
304L.....	FL4A	770	0.1	0.2	0.1
304L.....	FL3A	860	710[3]	....	0.1
304L.....	FL5A	730	670[3]	....	0.1
304L.....	FE5A	640	0.1	....	0.1

[1] Samples were activated in 15% HCl (by vol.) at 65 C before each test period. Samples were exposed for the indicated periods using fresh solution for each test period. A nonabrasive cleaning powder was used to remove any loose corrosion product on the samples at the end of each test period.
[2] A new set of samples was exposed for one 120-hour period in fresh solution without prior activation.
[3] These corrosion rates are based on a 24-hour exposure. Samples were removed from test after 24 hours in order to avoid their complete dissolution.

High manganese contents (15 to 17 percent) caused a significant decrease in the nitric acid corrosion resistance of the stainless steels containing 1 percent nickel or less. For example, Cr-Mn-N (17.5 to 18.0 percent Cr and 15.0 to 15.5 percent Mn) had a nitric acid corrosion rate 2 to 3 times that of Type 430 stainless steel (17.3 percent Cr-0.3 percent Mn). Other research has shown that the influence of manganese on the nitric acid corrosion resistance is much less pronounced at nickel contents of 4 percent or more.[29]

Boiling Phosphoric Acid. Activated tests in boiling 25 percent phosphoric acid revealed that the self-passivating tendencies of all three grades of lean-nickel steel in this environment were superior to those of Type 430 and Type 304 stainless steel (Table 15). The 120-hour rates for lean-nickel steels were 0.1 to 0.3 mil/mo. Tests on four heats of Type 304L stainless showed that when their natural passivity was destroyed by activation, they corroded at extremely high rates (650 to 850 mils/mo).

Air exposure of Type 304L following a period of severe corrosion restored the passivity in the case of two out of four heats. The pattern of corrosion behavior for Type 430 in activated tests was similar to that of Type 304L except that its rate of corrosion was much lower (7.5 mils/mo). Corrosion resistance of Type 430 and Type 304L was good in non-activated tests.

Nitrating Acid at 80 C. Corrosion resistance of Type 202 in nitrating acid (80 percent H_2SO_4-15 percent HNO_3-5 percent H_2O) at 80 C was comparable to that of Type 304L; 120-hour rates for the two types of steel were 0.7 to 0.8 mil/mo (Table 16). Corrosion rates for Type 201, Cr-Mn-N and TRC steel[8] were only slightly higher, 0.9 to 1.3 mil/mo. Type 430 (17 percent Cr) stainless had a corrosion rate more than twice that for Type 202.

Acetic Acid at 80 C. Type 304 and Type 202 stainless steel exhibited borderline passivity in 5 percent acetic acid at 80 C (Table 17). That is, under identical test conditions, samples of these steels would undergo active corrosion (11 to 15 mils/mo) in one case and not in another. Consequently, no conclusions can be drawn regarding the relative corrosion resistance of Type 202 and Type 304L in this environment.

None of the four very high manganese steels (Cr-Mn-N and TRC steel) were corroded in the three activated tests. In contrast, Type 430 underwent active corrosion in all three tests but tended to become passive upon air exposure or with continued test time.

Lactic Acid at 80 C. Type 201, Type 202 and Type 304L exhibited borderline passivity in 5 percent lactic acid at 80 C (Table 18). The resistance of the very high manganese steels to corrosion in nonactivated tests was superior to that of the other steels tested. This behavior suggests that manganese can contribute to stainless steel's passivity in nonoxidizing environments. Type 430 corroded severely in each of the three tests but became passive upon air exposure.

Formic Acid at 60 C. All of the stainless steels showed comparable resistance to corrosion in activated tests in 90 percent formic acid at 60 C (Table 19). In one test Type 430 became passive upon air exposure following a period of severe corrosion while the other steels did not. However, this behavior of Type 430 was not duplicated in additional tests.

Field Exposure in Nitric-Adipic Acid. Corrosion rate of Type 202L stainless steel field exposed for three months in 40 percent nitric-20 percent adipic acid solution at 95 C (Table 12) was about twice that of Type 304. Relative performance of these two types of steel in the field exposure was the same as that obtained in the boiling 65 percent nitric acid test (for the annealed condition). None of the other lean-nickel stainless steels was tested in this environment.

Summary of Test Results in the More Severely Corrosive Environments. These test results can be summarized by classifying the corrosive environments into three groups: (a) oxidizing environments, (b) nonoxidizing environments in which Type 304 corroded in a reproducible fashion and (c) non-oxidizing environments in which Type 304 exhibited borderline passivity.

(a) Oxidizing environments included boiling 30 percent and 65 percent nitric acid, 40 percent nitric-20 percent adipic acid solution at 95 C and nitrating acid

[8] This statement applies only to Heat HO1. Heat HD4 (TRC steel) showed erratic behavior in most of the corrosion tests involving nitric acid.

TABLE 16—Corrosion Data for Tests in Nitrating Acid at 80 C[3]

Type of Steel	Heat	CORROSION RATE, MIL/MO.[1] 1st Period, 24 Hour	2nd Period, 120 Hour	3rd Period, 24 Hour
430 (17% Cr)	GU8	2.5	1.8	2.9
Cr-Mn-N	HK7	1.8	1.1	1.0
Cr-Mn-N	HQ5	1.6	0.9	1.1
TRC	HD4	2.4	2.2	4.1[2]
TRC	HO1	2.1	1.3	1.3
201	GZ7	1.7	1.1	1.0
202	HO3	1.9	0.8	0.8
202	HN2	1.6	0.8	0.8
202L	HN3	1.7	0.7	0.7
202L	HP4	1.8	0.8	0.7
202L	HO4	1.7	0.8	0.5
304L	FL4A	1.8	0.8	0.7

[1] Samples were exposed for the indicated periods using fresh solution for each test period.
[2] HD4 underwent light intergranular attack.
[3] (80% H_2SO_4—15% HNO_3—5% H_2O).

TABLE 17—Corrosion Data for Activated Tests in 5% Acetic Acid at 80 C

Type of Steel	Heat Number	Number of Tests in Which Active Corrosion Occurred[1]	Range of Corrosion Rates, mil/mo.
430 (17% Cr)	GU8	3	0.3-22
Cr-Mn-N	HK7, HQ5	0	0
TRC	HD4, HO1	0	0
201	GZ7	0	0
202	HO3	0	0
202	HN2	2	8-14
202L	HN3, HP4, HO4	1	11-15
304L	FL4A	1	13

[1] Three activated tests of 24, 120, and 240 hours were made. Samples were electrolytically activated in test solution prior to each test. Fresh solution was used for each test. Samples from first test were reground and used for second test. A new set of samples was used for third test.

($80\%H_2SO_4$-$15\%HNO_3$-$5\%H_2O$) at 80 C. In the first three environments, which were the most strongly oxidizing in nature, the corrosion behavior was governed largely by the steel's nickel content. Type 201 and 202 stainless containing 4.5 to 5.5 percent Ni corroded at 1.5 to 2 times the rate of Type 304 (9.5 percent Ni). Cr-Mn-N and TRC steel containing 1 percent Ni or less corroded at a rate 3 to 9 times that of Type 304L.

In boiling 65 percent nitric acid, the very high manganese content of Cr-Mn-N and TRC steel resulted in a corrosion rate about triple that for Type 430 of a comparable chromium content.

In the nitrating solution, corrosion resistance of all four lean-nickel steel grades was, for practical purposes, comparable to that of Type 304. Corrosion rate for Type 430 in this environment was double that for Type 304.

(b) Nonoxidizing environments providing reproducible results included activated tests in 90 percent formic acid at 60 C and in boiling 25 percent phosphoric acid. In the formic acid environment, corrosion resistance of all stainless steels tested was comparable. In activated tests in boiling 25 percent phosphoric acid, the self-passivating tendencies of the four lean-nickel steel grades were superior to those of Type 430 and Type 304.

(c) Nonoxidizing environments involving borderline passivity were 5 percent acetic acid at 80 C (activated tests) and 5 percent lactic acid at 80 C (nonactivated tests). In general, Types 201 and 202 behaved similarly to Type 304 in these environments: all three exhibited borderline passivity. Type 430 corroded actively in each test but became passive upon subsequent exposure to air. Cr-Mn-N and TRC steels demonstrated a passivity superior to that of the other steels tested.

The lower nickel contents of the lean-nickel steels reduced their corrosion resistance in strongly oxidizing environments of a severely corrosive nature. However, corrosion resistance of the 4 percent minimum Ni grade, Type 202, was good enough to justify its consideration for such environments.

In certain nonoxidizing environments of a severely corrosive nature, high manganese contents contributed to the passivity of austenitic stainless steel and thus provided improved corrosion resistance.

TABLE 18—Corrosion Data for Nonactivated Tests in 5% Lactic Acid at 80 C

Type of Steel	Heat Number	Number of Tests in Which Active Corrosion Occurred[1]	Range of Corrosion Rates, mil/mo.
430 (17% Cr)	GU8	3	20–100
Cr-Mn-N	HK7, HQ5	1	0.1
TRC	HD4	2	0.1–1.4
TRC	HO1	0	0
201	GZ7	1	4
202	HO3, HN2	1	12–28
202L	HN3, HP4, HO4	1	16–65
304L	FL4A	2	42–43

[1] Three nonactivated tests of 24, 120 and 240 hours were made. Fresh solution was used for each test. Samples from first test were reground and used for second test. A new set of samples was used for third test.

Correlation Between Composition of Lean-Nickel Steels and Their General Corrosion Resistance. Of the four lean-nickel steel grades, Type 202 had the best resistance to general corrosion, due primarily to its higher minimum chromium content (17 percent) and higher minimum nickel content (4 percent) compared to the other Cr-Mn-Ni stainless alloys. The manganese content (7.5 to 10.0 percent) of Type 202 also contributed to its passivity in certain organic acids, such as acetic, oxalic and lactic, and in phosphoric acid. The individual contribution of Cr, Ni and Mn to the corrosion resistance of the lean-nickel stainless steels is discussed below.

Influence of Chromium Content. A minimum of 17 percent Cr is considered essential for adequate corrosion resistance in most chemical environments where Type 304 is used. Tests showed that in strongly oxidizing environments the corrosion resistance was impaired if the chromium content was reduced to 15 percent. (See corrosion data for TRC steel in Tables 11 and 14.)

Influence of Nickel Content. Nickel contributed markedly to the passivity of the steels tested, particularly in strongly oxidizing environments such as nitric acid. If the stainless alloy contained no nickel, passivity in organic acids such as formic and acetic was greatly reduced. (See corrosion data for Type 430 in Tables 5 and 7.)

Corrosion resistance of stainless alloys containing 17 percent Cr minimum in hot nitric acid solutions was directly related to the nickel content. For example, in boiling 65 percent nitric acid, ratio of corrosion rates for 0 percent Ni alloys (Type 430), 4 percent minimum Ni alloys (Type 202) and 9.5 percent Ni alloys (Type 304) was 3:2:1, respectively. Nickel exerted a similar influence on stainless steel's corrosion resistance in boiling 30 percent nitric acid and in 40 percent nitric-20 percent adipic acid at 95 C. A minimum of 4 percent Ni in the alloy probably will provide acceptable corrosion resistance for most plant applications involving hot nitric acid solutions; i.e., Type 202 stainless can be used.

Influence of Manganese. Manganese contributed to the self-passivating tendencies of stainless steel in corrosion tests made in three organic acids (acetic, lactic and oxalic) and in one mineral acid (phosphoric). In boiling 25 percent phosphoric acid and in 10 percent oxalic acid at room temperature, the capacity for self-passivation of all of the Mn-bearing stainless steels was superior to that of both Type 430 and Type 304. In 5 percent acetic acid and in 5 percent lactic acid (both at 80 C), the high Mn (15 percent) stainless alloys were completely passive while the other stainless steels exhibited borderline passivity.

When the Ni content of the stainless alloy is less than 4 percent, manganese contents greater than 10 percent can decrease the corrosion resistance in hot nitric acid solutions. For example, Cr-Mn-N (17 percent Cr-15 percent Mn) stainless steel showed higher corrosion rates in boiling 30 percent and 65 percent nitric acid than did Type 430 (17 percent Cr).

Pitting Tests.

Sodium Chloride Solution. A 4%NaCl solution (85 C) aerated at 50 to 75 ml/minute was used to test Type 430,

Cr-Mn-N, Type 201, Type 202, Type 202L and Type 304L stainless steel. These test conditions were used previously to study the pitting of 18-8 stainless steel.[37] Current tests consisted of one 24-hour test on specimens previously pickled in a HNO_3-HCl-HF solution and another 24-hour test on the same specimens in the as-ground condition. Neither test provided a satisfactory evaluation of the pitting resistance of the steels being studied. The first test did not produce sufficient pitting; the second test caused severe general corrosion, rather than localized pitting, on three of the six lean-nickel steels.

Ferric Chloride Solution. A 24-hour pitting test on pickled specimens in 5 percent ferric chloride solution at room temperature (Table 20) revealed the following: Type 430 stainless showed the greatest pitting tendency of the eight stainless steels tested. In addition, the Type 430 specimen was the only one which pitted on the flat faces. Pitting on the specimens of the other steels was confined almost completely to those edges which were perpendicular to the original rolling direction of the sheet or plate.

There were no observable differences in the pitting behavior of the lean-nickel grades (Cr-Mn-N and Types 201, 202, and 202L) of stainless steel and that of Type 304L stainless steel in this particular test. However, others[29] have reported that Type 202 stainless steel is not as resistant as Type 304 to localized attack in a salt-spray test.

Rusting Tests. Three tests were used to evaluate the relative rust resistance of Type 430, Cr-Mn-N, TRC steel, Type 201, Type 202, Type 202L and Type 304L stainless. These tests were (a) a 1000-hour exposure in a Weatherometer Unit operated at 40 C with a test cycle consisting of 9 minutes in a tap-water spray followed by a 51-minute drying period, (b) a 1000-hour exposure in a steam-sparging cabinet at 99 to 100 C and 100 percent relative humidity and (c) a four-month field exposure in a process dryer heated to 160 C to drive off water and traces of sulfuric acid from the product.

None of the stainless steels showed any practical difference in their rust resistance in the three tests. The weight losses were too small to be significant. Specimens of all of the steels were covered with an extremely light rust-colored stain after each test. Type 430 showed the greatest tendency to form individual rust spots, but even on this steel the rust spots were small and few in number.

Other investigators[3,17,38] have reported that the lean-nickel stainless steels have good resistance to atmospheric corrosion (or rusting). Atmospheric corrosion resistance of Type 201 in the New York and Pittsburgh areas was comparable to that of Type 304.[38] In these locations, TRC steel was resistant to pitting but underwent more rusting than Types 201 or 304.

TABLE 19—Corrosion Data for Tests in 90% Formic Acid at 60 C

Type of Steel	Heat Number	CORROSION RATE, MIL/MO. FOR ACTIVATED TESTS[1] 1st Period, 24 Hr.	2nd Period, 120 Hr.	3rd Period, 24 Hr.
430 (17% Cr)	GU8	2.2[2]	0	0
Cr-Mn-N	HK7	2.2	1.9	1.8
Cr-Mn-N	HQ5	2.6	2.2	2.8
TRC	HD4	3.5	2.8	3.9
TRC	HO1	3.0	2.5	3.8
201	GZ7	1.8	2.2	1.5
202	HO3	2.3	1.8	2.0
202	HN2	1.8	1.7	2.1
202L	HN3	2.2	1.8	2.6
202L	HP4	2.5	1.8	2.9
202L	HO4	2.2	1.9	2.4
304L	FL4A	2.4	2.3	2.7

[1] Samples were electrolytically activated in test solution prior to each test period. Samples were exposed for the indicated periods using fresh solution for each test period. A non-abrasive cleaning powder was used to remove any loose corrosion product on the samples at the end of each test period.
[2] GU8 was passive after first test period. This behavior of GU8 was not duplicated in additional tests, however. Activated tests on a new sample for a 120-hour period followed by a 24-hour period gave rates of 3.2 and 3.9 mil/mo., respectively.

TABLE 20—Corrosion Data for Pitting Tests in 5% Ferric Chloride at Room Temperature

Type of Steel	Heat Number	PITTING TEST RESULTS[1] Weight Loss, mg/sq dm/day	Number of Pits: Flat Faces	Longitudinal Edges[2]	Transverse Edges[3]
430 (17% Cr)	GU8	190	55	15	37
Cr-Mn-N	HK7	105	0	3	22
Cr-Mn-N	HQ5	70	0	1	26
201	GZ7	65	0	0	17
202	HO3	85	0	0	49
202L	HN3	70	0	0	31
202L	HO4	130	0	0	45
304L	FL4A	65	0	0	22

[1] Pitting test consisted of a 24-hour exposure in a 5% ferric chloride solution at room temperature. Prior to being tested, the samples were pickled in a solution containing 104 ml. of 65% HNO_3, 17 ml of 37% HCl, 17 ml of 48% HF, and 862 ml of H_2O at 80 C.
[2] Edges parallel to original rolling direction of the plate.
[3] Edges perpendicular to original rolling direction of the plate.

Intergranular Corrosion Tests.

Influence of Carbon Content. Intergranular corrosion resistance of the lean-nickel steels was evaluated in the boiling 65 percent nitric acid test for two of the most common sensitizing heat-treatments: 1 hour at 1250 F, WQ, and 2 hours at 1200 F, WQ. The results in Table 14 show that those lean-nickel steels having carbon contents greater than 0.03 percent had nitric acid corrosion rates for the sensitized condition of 0.0045 to 0.17 in./mo. These high rates were due to severe intergranular attack and heavy grain-dropping. The three heats of Type 202L (0.03 percent maximum C) did not undergo excessive intergranular attack in the nitric acid test; their rates for the sensitized condition were 0.0019 to 0.0027 in./mo.

The influence of carbon content on the degree of carbide precipitation in three heats of Types 202 and 202L is illustrated by the electrolytic oxalic acid etch structures in Figures 1-3.

Figure 4 graphically shows the differences in nitric acid corrosion behavior of these three heats. Data for curves in Figure 4 were obtained by converting the weight loss in grams per square centimeter for each 48-hour test period into the equivalent amount of metal lost in mils (corrosion was assumed to be uniform over the entire surface of the specimen). In the case of the 0.022 percent C heat (HP4), the sensitized specimen contained only minor amounts of grain-boundary carbides (see Figure 1B); consequently, it was not subject to intergranular attack in the nitric acid test. In Figure 4, the sensitized specimen of Heat HP4 corroded at the same rate as the annealed specimen. Sensitized specimens of the other Heats HN2 and HO3 underwent severe intergranular attack in the nitric acid test due to a grain-boundary network of precipitated carbides (see Figures 2B and 3B). As a result of the intergranular attack, sensitized specimens of Heats HO3 and HN2 were dropping grains from their surfaces after the first and third test periods, respectively. This grain-dropping is reflected in the increasing slope of the corrosion curves for the sensitized specimens of these two heats. The sensitized specimen of Heat HO3 with a more continuous network of grain-boundary carbides than the sensitized specimen of Heat HN2 was intergranularly corroding and dropping grains at a more rapid rate (note difference in ordinate scales of Figure 4 for Heats HN2 and HO3).

Comparison of Data. Information from concurrent investigations[27,29] indicated that the carbon content of the lean-nickel stainless steels was less critical than in the 18 percent Cr-8 percent Ni stainless steels. For example, it was claimed that 0.05 to 0.06 percent C Types 201 and 202 were as resistant to carbide precipitation upon sensitization as 0.03 percent maximum C Type 304.[29]

Figure 5 shows the influence of carbon



Figure 1—Electrolytic oxalic acid etch structures for Type 202 stainless steel (Heat HP4) containing 0.022 percent C. 130X. Part A: Specimen was commercially annealed; etch structure—step. Part B: Specimen was sensitized 1 hour at 1250 F, WQ; etch structure—mild dual.

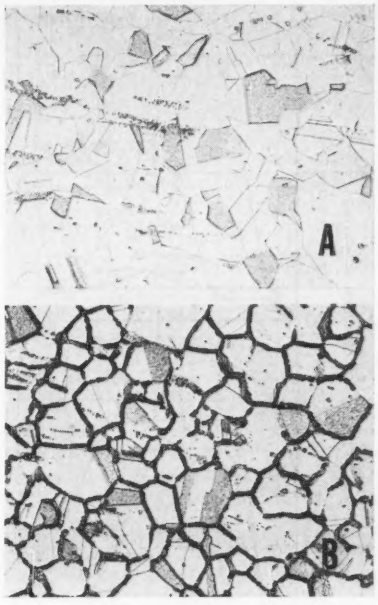


Figure 2—Electrolytic oxalic acid etch structures for Type 202 stainless steel (Heat HN2) containing 0.036 percent C. 130X. Part A: commercially annealed specimen with step etch structure. Part B: sensitized 1 hour at 1250 F, WQ; ditch (60 percent encircled grains) etch structure.

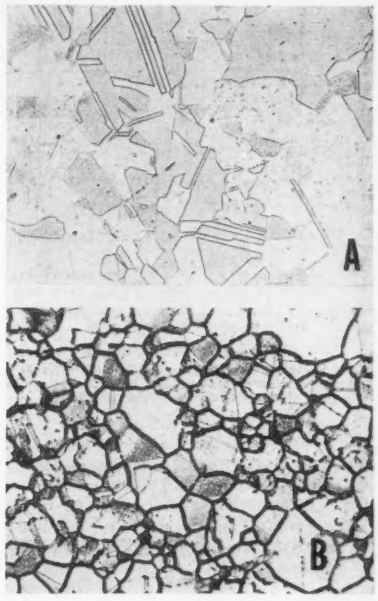


Figure 3—Electrolytic oxalic acid etch structure for Type 202 stainless steel (Heat HO3) containing 0.062 percent C. 130X. Part A: commercially annealed specimen with step etch structure. Part B: sensitized 1 hour at 1250 F, WQ, with ditch (95 percent encircled grains) etch structure.

TABLE 21—Effect of Composition (Mn + Ni Content) On Nitric Acid Corrosion Resistance of Type 202 Stainless Steel[5]

Heat Number	Carbon Content, %	Mn + Ni CONTENT OF LESS THAN 14.0		Mn + Ni CONTENT OF MORE THAN 14.0	
		Mn + Ni Content, %	Nitric Acid Corrosion Rate, in./mo.[1]	Mn + Ni Content, %	Nitric Acid Corrosion Rate, in./mo.[1]
DT87-3[4]	0.017	.....	.....	14.1	0.0017
HP4	0.022	.....	.....	15.4	0.0024
25350[4]	0.023	.....	.....	15.3	0.0020
HN3	0.030	.....	.....	14.5	0.0022
HN2	0.036	.....	.....	14.5	0.024
DG64[4]	0.040	12.4	0.0019	.....	.....
DT78A[4]	0.050	13.9	0.0022	.....	.....
HO3	0.062	.....	.....	14.7	0.13
DF76[4]	0.066	11.9	0.0027[2]	.....	.....
DT78B[4]	0.069	13.8	0.0046[3]	.....	.....

[1] Specimens were sensitized 2 hours at 1200 F, WQ, and flask-tested in boiling 65% nitric acid for five 48-hour periods. The average rate for the annealed condition was 0.0016 in./mo. (0.0014-0.0017 in./mo.).
[2] Fifth-period rate was 0.0041 in./mo. vs. 0.0015 in./mo. for first period; this indicates accelerated intergranular attack.
[3] Av. rate based on four periods instead of five.
[4] Data for these heats were taken from Reference 29.
[5] Steel was 0.017 to 0.069% C, sensitized 2 hours at 1200 F, WQ.

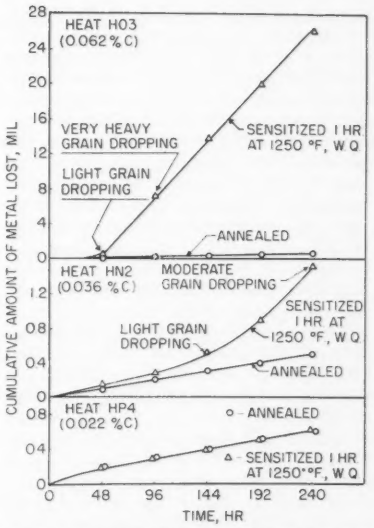


Figure 4—Corrosion of three Type 202 stainless steel heats of varying carbon content in boiling 65 percent nitric acid.

content on the nitric acid corrosion resistance of Type 202 versus Type 304 in the sensitized condition. The individual points represent the nitric acid corrosion data obtained in the current study for five commercial heats of Type 202. A 2-hour sensitizing treatment at 1200 F was used so that the current data could be compared with earlier research.[29]

Included in Figure 5 are curves showing the effect of carbon content on the nitric acid corrosion resistance of Type 304[(9)] and Type 202 as evaluated by Renshaw and Lula.[29] The sharp increase in the slope of the curves is due to severe intergranular corrosion and grain-dropping caused by damaging carbide precipitation. According to the curves, damaging carbide precipitation starts to occur in sensitized Type 304 at about 0.03 percent C and in sensitized Type 202 as evaluated by Renshaw and Lula at about 0.065 percent C. To the right of the dashed portion of each curve, the corrosion rate can vary over a wide range (0.002 to 0.2 in./mo) depending upon factors such as grain size and the continuity of the carbide network in the grain boundaries.

A comparison of the plotted points in Figure 5 with the accompanying curves shows that the intergranular corrosion behavior of the Type 202 heats in the current study is similar to that of Type 304. That is, when the carbon content of these Type 202 heats exceeded 0.03 percent, there was a sharp increase in corrosion rate caused by intergranular attack and grain-dropping (see corrosion rate for the 0.036 percent C Type 202 heat in Figure 5).

This apparent discrepancy between results of the current intergranular corrosion studies on Type 202 and results of previous studies[29] can be resolved, however. Binder, Thompson, and Bishop[27] found that the maximum carbon content for resistance of Type 202 to damaging carbide precipitation (upon heating

[(9)] Data used for the Type 304 curve were taken from Reference 39. However, similar results have been obtained by other laboratories.

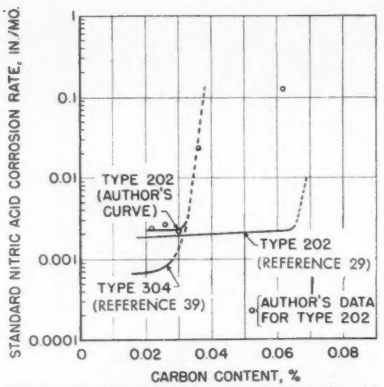


Figure 5—Influence of carbon content on nitric acid corrosion resistance of Type 202 vs Type 304 stainless steel sensitized 2 hours at 1200 F.

at 1200 F) decreased as the sum of the manganese and nickel contents increased above 14 percent. They also found that Type 202 stainless steel containing up to 0.05 percent C is free from damaging carbide precipitation after 1 hour at 1200 F if the Mn + Ni content does not exceed 14 percent.[27] Increasing the Mn + Ni content to 18 percent decreased the carbon limit to 0.035 percent.

The influence of Mn + Ni content on the current intergranular corrosion data and those of Lula and Renshaw[29] are shown in Table 21. The 0.04 and 0.05 percent C heats[29] which were resistant to intergranular corrosion after being sensitized 2 hours at 1200 F had Mn+Ni contents of 14 percent or less. In contrast, the 0.036 percent C heat (current study) which underwent severe intergranular attack after a similar sensitizing treatment had a Mn + Ni content of 14.5 percent. The influence of Mn + Ni content is also shown by the relative behavior of Heats DF76 and DT78B which have comparable carbon contents. Heat DT78B with a Mn + Ni content of 13.8 had a much higher nitric acid corrosion rate than heat DF76 with a Mn + Ni content of only 11.9.

Thus, the differences between the results of this study and those of other investigators[29] were due to differences in the Mn + Ni contents of the respective steels tested.

Influence of Heat-Treatment Time and Temperature. Effect of heat-treatment for 1, 2 and 24 hours at temperatures from 1000 to 1400 F was studied using four heats of Type 202 and 202L. Carbon contents of the four heats were 0.022 percent, 0.030 percent, 0.036 percent and 0.062 percent. The influence of heat-treatment on the nitric acid corrosion resistance is shown by the data in Table 22 and the curves in Figure 6. For a 2-hour heat-treatment, a 1200 F temperature was the most damaging to the nitric acid corrosion resistance. When the heat-treatment time was extended to 24 hours, temperature for maximum damage was shifted to 1100 F.

A 2-hour heat-treatment at temperatures from 1000 to 1400 F did not seriously damage the intergranular corrosion resistance of the 0.022 and 0.030 percent C heats of Type 202. However, a 24-hour

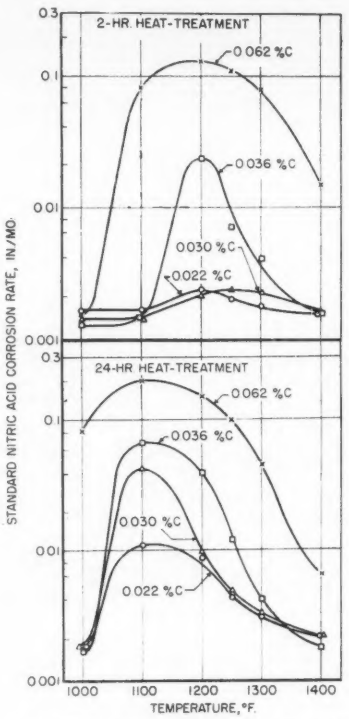


Figure 6—Nitric acid corrosion rates for Types 202 and 202L stainless steel heat-treated for 2 hours and 24 hours at indicated temperatures.

heat-treatment at temperatures in the range of 1050 to 1300 F resulted in severe intergranular corrosion of all four heats of Type 202 (0.022-0.062 percent C) in the nitric acid test. These results agree with other investigations that state, for Type 202 to be resistant to intergranular corrosion after heat-treatment for 24 hours at 1200 F, the maximum carbon content must be 0.020 percent if the Mn + Ni content is 14 to 18 percent.[27] All four Type 202 heats in this study had Mn + Ni contents over 14 percent and carbon contents over 0.020 percent.

Figure 7 shows that the influence of

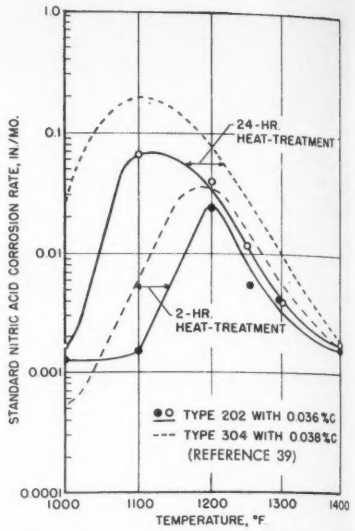


Figure 7—Influence of heat-treatment on nitric acid corrosion resistance of Type 202 vs Type 304 stainless steel.

heat-treatment on Type 202 having Mn + Ni contents of more than 14 percent is similar to the effect of heat-treatment on Type 304. Both 2-hour and 24-hour heat-treatments at temperatures from 1000 to 1400 F produced similar changes in the nitric acid corrosion resistance of the two types of steel. Comparison was made on the basis of two heats of steel having nearly equivalent carbon contents: 0.036 percent C for Type 202 vs. 0.038 percent C for Type 304.

Summary of Intergranular Corrosion Studies. The effect of sensitizing for 1 hour at 1250 F (or 2 hours at 1200 F) on the intergranular corrosion resistance of Type 202 is dependent on the sum of the Mn and Ni contents of the steel.

All Type 202 heats in the current studies had Mn + Ni contents of over 14 percent. Damaging carbide precipitation occurred when those heats contain-

TABLE 22—Effect of Heat-Treatment on the Nitric Acid Corrosion Resistance of Type 202 Stainless Steel of Varying Carbon Content

Temperature of Heat-Treatment, F	Time of Heat-Treatment, Hr.	CORROSION RATE, IN./MO. FOR STANDARD NITRIC ACID TEST[1]			
		Heat HP4 (0.022% C)	Heat HN3 (0.030% C)	Heat HN2 (0.036% C)	Heat H03 (0.062% C)
1000	1	0.0017	0.0015	0.0014	0.0014
	2	0.0018	0.0014	0.0013	0.0015
	24	0.0019	0.0019	0.0017	0.083
1100	1	0.0016	0.0014	0.0013	0.026
	2	0.0017	0.0015	0.0015	0.083
	24	0.011	0.043	0.067	0.20
1200	1	0.0019	0.0019	0.0081	0.12
	2	0.0024	0.0022	0.024	0.13
	24	0.0087	0.0098	0.040	0.15
1250	1	0.0019	0.0023	0.0045	0.078
	2	0.0021	0.0024	0.0062	0.11
	24	0.0043	0.0049	0.012	0.096
1300	1	0.0018	0.0020	0.0035	0.063
	2	0.0018	0.0023	0.0042	0.079
	24	0.0032	0.0033	0.0041	0.046
1400	1	0.0016	0.0015	0.0017	0.011
	2	0.0016	0.0017	0.0016	0.015
	24	0.0022	0.0022	0.0018	0.0065

[1] Based on individual flask tests in boiling 65% nitric acid for five 48-hour periods.

TABLE 23—Tensile Properties of Butt-Welded Joints of Cr-Mn-N, Type 202, and Type 202L Stainless Steel Compared to Those of Type 304L

Type of Steel	Heat Number	Sheet or Plate Thickness, in.	TENSILE PROPERTIES OF WELDED JOINTS					
			Metallic-arc Process		Tungsten Inert-arc Process		Average Properties for Both Processes	
			Ult. Tensile Strength, psi	Percent Elongation in 2 in.	Ult. Tensile Strength, psi	Percent Elongation in 2 in.	Ult. Tensile Strength, psi	Percent Elongation in 2 in.
Cr-Mn-N	HQ5	0.170	112,500 120,500	13 17	118,000 118,500	20 17	117,500	17
202	HO3	0.230	100,000 104,500	24 31	99,000 99,000	26 29	100,500	28
202	HN2	0.060	85,500 90,500	..[2] 27	103,000 103,500	20 37	95,500	28
202L	HO4	0.280	98,000 99,500	30 29	95,500 101,500	28 26	98,500	28
202L	HN3	0.060	75,500 95,500	..[2] 18	105,500 98,000	22 22	94,000	21
304L[1]	FL3A	0.190	83,500 76,500	33 34	85,500 87,500	44 45	83,000	39

[1] Heat FL3A is a high nitrogen (0.13%) heat of Type 304L stainless steel. Consequently, the tensile strengths reported here may be higher than those usually obtained from Type 304L.
[2] Elongation is not listed because of low value resulting from eccentric loading in the tensile test. The butt welds in the lighter gage material frequently caused specimen distortion which in turn led to eccentric loading.

TABLE 24—Nitric Acid Corrosion Rates for Welded and Welded-and-Annealed Samples of Types 202 and 202L Stainless Steel

Type of Steel	Heat Number	Carbon Content, %	CORROSION RATE, IN./MO. FOR STANDARD NITRIC ACID TEST[1]			
			Tungsten Inert-Arc Welded		Metallic Arc-Welded	
			As-Welded	Welded and Annealed[2]	As-Welded	Welded and Annealed[2]
202	HO3	0.065	0.0012	0.0011	0.0012	0.0012
202	HN2	0.036	0.0014	0.0012	0.0012	0.0013
202L	HN3	0.030	0.0014	.....	0.0013	.....
202L	HO4	0.026	0.0014	0.0014	0.0013	0.0013
304L	FL4A	0.026	0.00079	.....	0.00088	.....

[1] Based on individual flask tests in boiling 65% nitric acid for five 48-hour periods.
[2] Welded samples were annealed 30 minutes at 1950 F, WQ.
Note: After the nitric acid test, none of the samples showed evidence of weld decay (severe intergranular attack and grain dropping in the heat-affected zone) or of excessive corrosion of the weld metal.

ing more than 0.03 percent C were sensitized for 1 to 2 hours at 1200 to 1250 F. Those heats containing 0.03 percent maximum C were resistant to intergranular corrosion in the standard nitric acid test after heat-treatment for 2 hours at 1200 F but not after 24 hours at 1200 F.

Heat-treatment from 1000 to 1400 F had a similar influence on the intergranular corrosion behavior of Types 304 and 202 stainless steel having a Mn + Ni content of over 14 percent.

Weldability Studies

Reported below are results of weldability studies on Cr-Mn-N, Type 202, Type 202L and Type 304L stainless steel. Table 4 lists the welding conditions used.

General Welding Characteristics. Welding characteristics of the lean-nickel steels were normal and were comparable to those for the 18 percent Cr-8 percent Ni grades of austenitic stainless steel. No unusual difficulties were experienced with arc control, undercut or spatter.

Radiographic Examination. All welds were of good quality and free from slag inclusions, hot tears and excessive porosity. Even the high nitrogen steel (Cr-Mn-N) was surprisingly free from weld metal porosity.

Bend Tests. Welds of all lean-nickel steels displayed good ductility in the face and root bend tests. There was no evidence of cracking, even in the presence of sharp notches. (Some notches were unavoidably present because the undercut on some specimens of lighter gage material could not be removed without seriously reducing the cross section.)

Tensile Tests. Tensile properties of the welded lean-nickel steels were quite good as shown by the data in Table 23. Tensile strengths for welded joints of Types 202 and 202L stainless steel ranged from 95,000 to 100,000 psi; elongations ranged from 21 to 28 percent in 2 inches. Welded joints of Cr-Mn-N stainless steel were slightly higher in tensile strength (118,000 psi) and slightly lower in ductility (17 percent). These mechanical properties for the lean-nickel steels can be compared with those given by ASTM Specification A-240-54 for unwelded Type 304 stainless steel plate: tensile strength of 75,000 psi (min.) and elongation of 30 percent (min.).

Results of the current investigation were comparable to the results of other weldability studies[24] on Types 201, 202, and 202L. Prior studies showed tensile strengths of 90,000 to 108,000 psi and elongations of 19 to 34 percent for butt-welded joints made by the metallic-arc process or by the tungsten inert-arc process with filler metal additions.[24]

Summary of Weldability Studies. Weldability of Cr-Mn-N, Type 201 and Type 202 by the metallic-arc or tungsten inert-arc process was good. Tensile strengths of butt-welded joints of these materials were higher than those of Type 304.

Nitric Acid Testing of Welded Samples

Specimens of Types 202, 202L and 304L welded by the metallic-arc and tungsten inert-arc processes were subjected to the boiling 65 percent nitric acid test. Nitric acid corrosion rates for the as-welded and welded-and-annealed specimens were comparable to those for the unwelded base plate (see data in Table 24).

None of the as-welded specimens showed any evidence of preferential attack on the weld metal or the heat-affected zone after nitric acid testing. Absence of intergranular attack and grain-dropping in the heat-affected zone does not mean that Type 202 containing more than 0.03 percent C cannot be sensitized by welding. Another investigator[24] using a 15 percent nitric-3 percent hydrofluoric acid test at 60 C found that Types 201 and 202 stainless steel were sensitized by welding but Type 202L (0.03 percent maximum C) was not.

Summary

Corrosion Resistance

The general corrosion resistance of four grades of lean-nickel austenitic stainless steel was evaluated in over 20 different environments ranging from mildly to severely corrosive in nature. These four grades of stainless steel were Cr-Mn-N, TRC steel, Type 201 and Type 202.[10]

Types 430 and 304[10] were included in the corrosion evaluation as standards of comparison. Results of the corrosion evaluation were as follows:

Mildly-to-Moderately Corrosive Environments. The corrosion resistance of

[10] See Footnote 6.

Type 202 was equivalent to that of Type 304 in all environments of this type. These environments included all types of cold organic acids, strongly oxidizing acids at temperatures up to 60 C, and environments which produced rusting. Corrosion resistance of Cr-Mn-N stainless steel in these environments was good, but in some cases it was not equivalent to that of Type 202. TRC steel was inferior to the other lean-nickel steels because of its lower chromium content.

More Severely Corrosive Environments. In non-oxidizing environments of a severely corrosive nature, corrosion resistance of Type 202 was equal to that of Type 304. In some cases it was even superior.

In strongly oxidizing environments such as boiling 30 percent and 65 percent nitric acid, corrosion resistance of Type 202 was inferior to that of Type 304 but was still within the useful range (corrosion rates of 0.0015 in./mo. or less).

Intergranularly Corrosive Environments. The intergranular corrosion resistance of Type 202L (0.03 percent maximum C) was comparable to that of Type 304L (0.03 percent maximum C) when both steels were heat-treated for 1 hour at 1250 F, WQ.

Weldability and Strength

Weldability of two lean-nickel stainless steel grades was evaluated for the metallic-arc and tungsten inert-arc welding processes, including radiographic examination, guided bend tests and tensile tests on butt-welded samples. Weldability of Cr-Mn-N and Type 202 stainless steel was good and was comparable to that of Type 304. (TRC steel and Type 201 were not included in the weldability studies.) Tensile strengths of butt-welded joints of the lean-nickel steels were appreciably higher than those of Type 304.

Conclusions

The following conclusions can be drawn from the above results. Type 202 (0.10 percent maximum C) stainless steel can be used for most chemical plant applications where Type 304 is giving satisfactory service. In the case of severely corrosive environments, field or laboratory tests should be used to determine if Type 202 would have adequate corrosion resistance for the particular application involved.

Type 202L can be used safely in the as-welded condition in those environments where there is danger of intergranular corrosion.

Acknowledgments

Particular thanks are due J. R. Auld and H. M. High for help in outlining the corrosion test program and in making the field exposures.

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Any discussion of this article not published above will appear in June, 1960 issue.

NATIONAL ASSOCIATION of CORROSION ENGINEERS

Section Three

Bibliographies of Corrosion Products

By NACE Technical Unit Committee T-3B on Corrosion Products★

NACE TECHNICAL COMMITTEE REPORT
Publication 60–4

Abstract

Selected abstracts are given on the identification and composition of corrosion products on cobalt and cobalt alloys, copper and copper alloys, chromium and chromium alloys, nickel and nickel alloys, and molybdenum and molybdenum alloys. Included are 15 abstracts on cobalt; 16 on copper; 13 on chromium; 16 on nickel; and 19 on molybdenum. 1.4

Introduction

BIBLIOGRAPHIES in this section were prepared by Technical Unit Committee T-3B on Corrosion Products. This work, the third in a series of technical committee reports,[1] is an introductory reference to the subject of "corrosion products" and is not intended to be exhaustively complete. Selected key references and some others of particular interest are given, omitting many which probably might well have been included.

All members of Committee T-3B contributed. E. L. Simons assisted in coordinating work of the contributors and M. C. Bloom, E. L. Simons, H. P. Godard, N. A. Nielsen and R. T. Foley edited the material.

Part 11—COBALT AND COBALT ALLOYS*

Corrosion Products

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The Kinetics of the Oxidation of Cobalt. EARL A. GULBRANSEN AND KENNETH F. ANDREW. *J. Electrochem. Soc.,* **98,** No. 6, 241-251 (1951). The problem is complex, since two or more oxides are formed and the metal itself has a phase transition from h.c.p. to f.c.c. The vacuum-microbalance method was used to study the effect of time, temperature, pressure, pretreatment, etc., on the rate of the reaction below 700 C. The reaction of cold-worked Co (h.c.p. form) and annealed Co. (largely f.c.c. form) was studied over the range 200-750 C, and it was found that colored oxides were formed at all temperatures except 200 C. In the range 600-700 C, the oxide films cracked on cooling, whereas below 600 C they appeared adherent. The cold-worked samples oxidized far more readily than the annealed ones. The results show that the reaction: $Co_3O_4 + Co \rightarrow 4CoO$ occurs readily at 450 C.

On the Oxidation of Cobalt at High Temperatures. GABRIEL VALENSI. *Metaux et Corrosion,* **25,** No. 303, 283-291 (1950). A discussion of the results of experiments on dry oxidation of cobalt, and on equilibrium between CoO and Co_3O_4.

The Anodic Behavior of Metals. VI. Cobalt. S. E. S. EL WAKKAD AND A. HICKLING. *Trans. Faraday Soc.,* **46,** No. 10, 820-824 (1950). In alkaline solutions, in which passivity is almost at once set up, three stages of oxidation have been observed corresponding to the formation of CoO, Co_2O_3, and CoO_2.

Oxide Films Formed on Alloys at Moderate Temperatures: Electron-Diffraction and Electron-Microscope Study. EARL A. GULBRANSEN, R. T. PHELPS AND J. W. HICKMAN. *Indust. and Eng. Chem.* (Analyt. Edn.), **18,** No. 10, 640-652 (1946). Electron-microscope and electron-diffraction data are given for electrochemically and chemically stripped films from alloys consisting principally of iron, cobalt, nickel, and chromium, cobalt-iron alloys and Kovar. Simple oxides of cobalt are not formed.

Comparative Study of the Anodic Oxidation of Nickel, Cobalt, and Manganese in Alkaline Solutions. JEAN BESSON. *Compt. Rend.,* **223,** No. 1, 28-30 (1946). In the case of cobalt, there are two points of inflection $CoO_2 = Co_2O_3$, and $Co_3O_4 = CoO$.

Texture in Cobalt and Nickel Tarnish. V. I. ARKHAROV AND G. D. LOMAKIN. *J. Tech. Physiks,* (USSR), **14,** 151 (1944). Below 890 C, the scale consists of a thin surface layer of Co_3O_4 and a thicker layer of CoO underneath, while above 890 C only CoO is present.

An Accurate X-ray Investigation of the Oxides of Iron, Cobalt, and Nickel. V. I. ARKHAROV AND K. M. GRAEVSKY. *Zhur. Tekhn. Fiziki,* **14,** No. 3, 132-145

★A. H. Roebuck, The Western Company, Ft. Worth, Texas, chairman.

[1] Section One of "Bibliographies of Corrosion Products" appeared in *Corrosion,* **13,** 111t (1957) Feb. Section Two appeared in *Corrosion,* 13, 565t (1957) Sept.

* Prepared by F. R. Morral, Battelle Memorial Institute, Columbus, Ohio.

(1944). In the case of CoO, the lattice constant increases towards the outside.

On the Structure of Oxides of Cobalt and Nickel. V. I. ARKHAROV AND G. D. LOMAKIN. *Zhur. Tekhn. Fiziki*, **14,** No. 3, 155-161 (1944). The oxide film on cobalt consists of an internal layer of CoO and a thin external layer of Co_3O_4 below 890 C, while above 890 C the oxide film consists of CoO. In CoO indications of a structure having (110) planes parallel with the surface were obtained at temperatures below 850 C. At 850-930 C an additional structure appears with the planes (601) parallel to the surface. At 930 C only the second type of structure remains.

Velocity of Oxidation of Cobalt. G. CHAUVENET AND G. VALENSI. *Comptes Rendues,* **205,** 317-319 (1937). Find Co_3O_4 below 750 and CoO above 850 C, and a mixture between these temperatures.

X-ray Investigation on the High-Temperature Oxidation of Co. V. I. ARKHAROV AND Z. A. VOROSHILOVA. *Zhur. Phys. Chem.* A, **142,** 151-175 (1929). Cobalt heated in air between 385-800 C has scale consisting largely of CoO in contact with metal and a small amount of Co_3O_4 is greater at the lower temperature.

Part 12—COPPER AND COPPER ALLOYS*

Corrosion Products

The Surfaces of Copper Alloys by Electron Diffraction. H. TAZAKI AND H. HASHIMOTO. *J. Sci. Hiroshima Univ.*, Ser. A, **14,** 44-7 (1949). Four commercial brasses were exposed to air at room temp., heated to 200-950 C, heated in an oxidizing gas flame, and exposed to HCl, HNO_3, and H_2SO_4. Various oxides formed when the specimens were heated at 200-400 C and 500-900 C were discussed. (*C.A.*, **47,** 5858b (1953).)

A Study of Oxide Films Formed on Copper-Magnesium Alloys by Electron Diffraction. H. TAZAKI AND S. KUWABARA. *J. Sci. Hiroshima Univ.*, Ser. A, **15,** 67-70 (1951). At temperatures from 0-250 C and air pressures of 10^{-2} mm-100 mm only Cu_2O could be detected. CuO was also found at the higher pressures if temperatures exceeded 250 C. The samples studied contained 1, 3, 5, and 10 percent Mg. At pressure exceeding 0.1 mm and temperature exceeding 350 C MgO was found to be present. At high temperatures and atmospheric pressure the film was found to consist of 3 layers—CuO at the surface, MgO in the middle layer, and Cu_2O adjacent to the oxide-alloy interface. (*C.A.*, **47,** 5337h, (1953).)

Oxide Films Formed on Copper-Aluminum Alloys by Electron Diffraction. H. TAZAKI AND S. KUWABARA. *J. Sci. Hiroshima Univ.*, Ser. A, **15,** 133-7 (1951). Alloys studied contained 2, 6, and 10 percent Al. CU_2O only could be identified at low pressures and temperatures up to 550 C. γ-Al_2O_3 and $CuAl_2O_4$ were detected at temperatures over 575 C. (*C.A.*, **47,** 5858c (1953).)

The Production of Lower Oxides on the Air-Chilled Surface of Horizontally Cast Copper Wire Bars. P. KLARE. *Z. Erzbergbau u. Metallhuttenw.*, **5,** 350-3 (1952). Cu_2O was found on the air-chilled surface. The mechanism of formation is presented. (*C.A.*, **47,** 85f (1953).)

High Temperature Oxidation Characteristics of a Group of Oxidation-Resistant Copper-Base Alloys. J. P. DENNISON AND A. PREECE. *J. Inst. Metals,* **81,** 229-34 (1953) (Paper No. 1436). The effects of Al, Be, Cr, Mg, and Si on oxidation resistance were determined. Be gave best resistance. Al, Mg, Si, and Cr follow in that order of effectiveness. (*C.A.*, **47,** 2108f (1953).)

The Mechanism of Pitting Corrosion. R. MAY. *J. Inst. Metals,* **82,** 65-74 (1953) (Paper No. 1497). Mechanism, necessary conditions for pit formation, and nature of corrosion products on Cu are discussed. (*C.A.*, **48,** 532a (1954).)

Electron Microscope Study of the Oxidation Process of Metals. G. PFEFFERKORN. *Naturwissenshaften*, **40,** 551-2 (1953). Incipient oxidation in the form of needle growth on Cu, Zn, Ni, Mo, and Fe surfaces is presented in photographs at 11,300 X. The needle melting point is below the oxide melting point. Growth occurs at needle ends. (*C.A.*, **48,** 8144h (1954).)

Differentiation of Cuprous Oxide and Cuprous Sulfide on Copper Micrographs. L. A. BOSCHI, H. DESTAILLATS, J. A. SABATO, J. M. VALLS, AND A. VARAVSKY. *Rev. Met.*, **51,** 774-6 (1954). Description of electropolishing technique. Electrolyte is H_3PO_4. (*C.A.*, **49,** 2274c, (1955).)

Attack of Various Superheated Steam Atmospheres Upon Aluminum Bronze Alloys. A. P. C. HALLOWES AND E. VOCE. Based on B.N.F.M.R.A. Report R.R.A. 527, Pt. II. *Metallurgia,* **34,** No. 201, 119-122 (1946) July. Failure of hollow truncated DTD197 (10Al-5Fe-5Ni) bronze cones used as plungers in manufacture of glass tumblers, and of Fe-Mn Bronze valve bodies controlling a superheated steam supply led to experiments. Tests were made with 10, 11, 12, 2, 5 Al bronzes. DTD197 bronze, 60-40 brass, 88-10-2 and 90-10 Sn- bronzes in pure steam and steam contaminated with various concentrations of SO_2 and Cl_2 at various temperatures and for varying lengths of times. Pure steam was harmless. Prolonged exposure to steam contaminated with either Cl_2 or SO_2 at elevated temperature produced attack on all but the 60-40 brass. Similar results were obtained with tap and distilled water. Nature of corrosion products is described and microstructures are shown.

F. DE CARLI AND N. COLLARI. *Metallurgia Italiana,* **54,** 1 (1952). Studies of the oxidation of bronzes with 6-37 percent Sn at 500-800 C. For alloys with 6 percent tin, the ratio of Sn to Cu in oxide was less than in metal. For higher contents of tin, the ratio of Sn to Cu was greater in the oxide.

W. D. CLARK. *J. Inst. Metals,* **73,** 263 (1947). Phosphor-bronze (11 percent tin) in hot water was "destannified," the corrosion product consisting mainly of metallic copper and stannic oxide.

S. C. BRITTON. *J. Inst. Met.*, **67,** 119 (1941). Two percent tin bronze in sheltered exposure to atmosphere highly polluted with coal smoke produced corrosion product consisting mainly of basic sulfate and chloride of copper. Tin was present in product, but compound was not identified.

W. L. COLLINS. *J. Inst. Met.*, **45,** 23 (1931). Ancient Chinese Bronzes after earth burial are covered with a mixture of copper minerals (cuprite, malachite, azurite, etc.) with stannic oxide occurring in pockets.

C. F. ELAM. *J. Inst. Met.*, **48,** 97 (1932). Ancient Bronzes from Ur had outer layer of copper mineral (believed to be mainly malachite) with inner layer of cuprite into which tin and nickel appeared to enter.

R. J. GETTENS. *J. Chem. Education,* **28,** 67 (1951). Corrosion products on ancient Chinese Bronzes (20 percent Sn and 4 percent Pb) are in layers with first (inner) residual dendritic metal with cupric and stannic oxides. Then cuprous chloride, redeposited copper, cuprous oxide, and stannic oxide and finally basic copper chloride and carbonate.

R. WOOD AND G. H. WYATT. *J. Soc. Chem. Ind.*, **62,** 110 (1943). Phosphor-bronze springs in electrical apparatus developed basic copper carbonate and tin oxide by action of ozone, carbon dioxide, and moisture.

Part 13—CHROMIUM AND CHROMIUM ALLOYS**

Corrosion Products

The Structure of Oxide Scales on Chromium Steel. H. J. YEARIAN, E. C. RANDELL AND T. A. LONGO. *Corrosion,* **12,** 10, 515-525t (1956) October. The structure of the oxide scale formed on commercial chromium steels containing 5 to 26 percent chromium was determined by X-ray diffraction, supplemented by chemical analysis.

High Temperature Scaling of Cobalt-Chromium Alloys. C. A. PHALNIKAR, E. B. EVANS AND W. M. BALDWIN, JR. *J. Electro Chem. Soc.*, **103,** 8, 429-438 (1956) August. Scaling rates and scale

* Prepared by R. C. Schueler, Phillips Petroleum Company, Bartlesville, Oklahoma.

** Prepared by T. M. Swain, Technical Service Department, Solvay Process Division, Allied Chemical Corp., Syracuse, N. Y.

compositions of cobalt-chromium alloys were determined in the temperature range of 900-1200 C.

Radiochemical Analysis for Iron, Chromium, Nickel, and Cobalt Corrosion Products. R. R. SMITH, T. O. PASSELL AND S. D. REEDER. U. S. Atomic Energy Comm., Pubn. AECD, 3889 (1955) September 9. The determination of corrosion products of Inconel is described.

Effects of High Temperature Oxidation on the Surface Composition of a Nickel-Chromium-Titanium-Aluminum Alloy. (In French.) FRIMA MALAMAND AND GEORGES VIDAL. *Comp. rend.*, **240,** 186-188 (1955). January 10. Compositions at the surface and at various depths in a Nichrome type alloy after high temperature oxidation in air were determined spectrographically.

Nitriding of Austenitic Chromium-Nickel-Molybdenum Steel Tubes by Nitrogen in Internal Pressure Tests at 600 C. F. BRAUMANN AND N. KRACHTER. *Arch. Eisenhuttenw.*, **25,** 373-375 (1954) July/August. Chemical, metallographic and X-ray investigations on the nitrided layer on the inner surface of the tubes are given.

Comparative Study of the Mechanism of the Oxidation of Binary Alloys Iron-Chromium, Nickel-Chromium, and Nickel-Aluminum. (In French.) JEAN MOREAU AND JACQUES BENARD. *Comp. rend.*, **237,** 22, 1417-1419 (1953). Surfaces of specimens oxidized in air at 800-1300 C were examined by X-ray diffraction and micrography.

Oxide Film on Nickel-Chromium Alloy Crystal Structure Studies. E. A. GULBRANSEN AND W. E. MCMILLAN. *Ind. Eng. Chem.*, **45,** 8, 1734-1744 (1953) August. Crystal structure studies were made on oxide films of eight alloys of nickel-chromium using electron diffraction methods.

Study of the Mechanism of the Oxidation of Binary Alloys of Iron-Chromium at Elevated Temperatures. JEAN MOREAU. *Comp. rend.*, **236,** 85-87 (1953) January 5. The oxides of iron-chromium alloys formed at 800-1200 C were examined by means of X-ray diffraction and micrography.

High Temperature Oxidation of Some Iron-Chromium Alloys. D. CAPLAN AND M. COHEN. *J. Metals*, **4,** 10, 1057-1065 (1952) October. Scaling characteristics of these alloys were investigated. The scales obtained were examined by X-ray diffraction and by spectrographic and metallographic analysis.

Oxide Films Formed on Alloys at Moderate Temperatures. EARL A. GULBRANSEN, R. T. PHILLIPS AND J. W. HICKMAN. *Ind. Eng. Chem.*, **18,** 640-652 (1952) October. Electron microscope and electron diffraction data are presented on the structure of electrochemically and chemically stripped films on alloys of iron, cobalt, nickel and chromium.

Passivity of Chromium. NORMAN HACKERMAN AND D. I. MARSHALL. *J. Electrochem. Soc.*, **93,** 49-54 (1948) March. Surfaces of chromium, treated with nitric acid and then oxidized in air were examined by electron diffraction.

An Electron Diffraction Study of Oxide Films Formed on High Temperature Oxidation Resistant Alloys. J. W. HICKMAN AND E. A. GULBRANSEN. Electrochem. Soc. Reprint 91-32, 467-483 (1947). Electron diffraction reflection is used to study the structures of oxides formed on high chromium alloys.

An Electron Diffraction Study of Oxide Films Formed on Iron, Cobalt, Chromium and Copper at High Temperatures. E. A. GULBRANSEN AND J. W. HICKMAN. *Metals Tech.*, **13,** 7, 1-26 (1947) October. An electron diffraction study of the oxide films on iron, cobalt, chromium and copper.

PART 14—MOLYBDENUM AND MOLYBDENUM ALLOYS*

Corrosion Products

The Electrochemical Oxidation of Molybdenum in Potassium Hydroxide Solutions. M. DEKAY THOMPSON AND A. L. KAYE. *J. Electrochem. Soc.*, **62,** 255-65 (1932). The oxidation products, soluble molybdate, greenish $Mo(OH)_4$ and colloidal blue Mo_3O_8 are reported.

The Corrosion Resistance of Metals to Nitric Acid Solution. Y. YAMAMOTO. *Bull. Inst. Phys. Chem. Research* (Tokyo), **19,** 281-366, 437-88, 587-713 (1940). A stable oxide coating formed on Mo and was protective against further attack by HNO_3. [*C.A.*, **35,** 2460[9] (1941).]

Electron Diffraction and Electron Microscope Study of Oxide Films Formed on Metals and Alloys at Moderate Temperatures. R. T. PHELPS, E. A. GULBRANSEN AND J. W. HICKMAN. *Ind. and Eng. Chem.*, Anal. Ed., **18,** 391-400 (1946). Mo was oxidized in 0.1 atmosphere of oxygen at 450 and 500 C. Mo_2O_3 was reported for 450 C, and Mo_2O_3 and MoO_3 were reported for 500 C. Later work by the authors shows that oxide believed to be Mo_2O_3 was MoO_2.

A Picturesque Oxidation of Molybdenum Vacuum-Tube Leads. H. A. LIEBHAFSKY. *J. Applied Phys.*, **17,** 901-2 (1946). Needles of MoO_3 were formed on Mo at 580 C in a nitrogen atmosphere which contained oxygen and water vapor. X-ray diffraction was used to identify MoO_3.

Dissociation Pressures of Metal Oxides Formed on Various Solid Metals. B. LUSTMAN. *Steel Processing*, **32,** 669, 676 (1946). Dissociation pressures up to the melting point and extrapolations to the boiling point are given for the oxides formed on Mo. [*C.A.*, **41,** 332h (1947).]

* Prepared by J. W. Semmel, Jr., Metallurgy and Ceramics Research Dept., General Electric Company, Schenectady, N. Y.

An Electron Diffraction Study of Oxide Films Formed on Molybdenum, Tungsten, and Alloys of Molybdenum, Tungsten, and Nickel. J. W. HICKMAN AND E. A. GULBRANSEN. *Trans. Am. Inst. Mining Met. Engrs.*, **171,** 371-88 (1947). The oxide films formed on Mo and Mo alloys between 300 and 700 C in 1 mm pressure of oxygen were studied by high-temperature electron diffraction. MoO_2 and MoO_3 were observed, and the temperatures at which one or both of the oxides occur were determined.

Electropotentials in Growing Halide and Oxide Layers on Metals. A. DRAVNIEKS AND H. J. MCDONALD. *J. Electrochem. Soc.*, **93,** 177-90 (1948). The oxide on Mo was studied by the electrode probe method during its formation in air up to 400 C. The oxide was predominately an electronic conductor.

Investigations of Gas-Metal Reactions by Reflection Electron Diffraction. J. W. HICKMAN. *Pittsburgh Intern. Conf. on Surface Reactions*, 142-56 (1948). High-temperature electron diffraction patterns were obtained from oxides formed on Mo and Mo alloys in 1 mm pressure of oxygen at 300-700 C. MoO_2 and MoO_3 were observed, and the temperatures at which one or both of the oxides occur were determined.

Thin Oxide Films on Molybdenum. E. A. GULBRANSEN AND W. S. WYSONG. *Trans. Am. Inst. Mining Met. Engrs.*, **175,** 628-47 (1948). Protective MoO_2 formed at elevated temperatures up to 1000 C in vacuum on the order of 10^{-6} mm of Hg. The MoO_2 was identified by electron diffraction at 900 C. Oxidation was studied at 250-450 C.

Oxide Films Formed on Metals and Binary Alloys. An Electron Diffraction Study. J. W. HICKMAN. *Trans. Am. Inst. Mining Met. Engrs.*, **180,** 547-64 (1949). The oxide films formed on Mo and Mo alloys between 300 and 700 C in 1 mm pressure of oxygen were studied by high-temperature electron diffraction. MoO_2 and MoO_3 were observed, and the temperatures at which one or both of the oxides occur were determined.

Volatility of the Oxides of Tungsten and Molybdenum in the Presence of Water Vapor. T. MILLNER AND J. NEUGEBAUER. *Nature*, **163,** 601-2 (1949). The presence of water vapor in one atmosphere of oxygen increases the volatility of MoO_3 at 600 and 700 C.

Oxidation of Molybdenum in Air at 1100 to 1600 F. B. LUSTMAN. *Metal Progress*, **57,** 629-30, 674 (1950). The volatility of MoO_3 was investigated by determining the fraction of oxidized metal remaining as adherent oxide. The weight change method was used.

The Formation of Molybdenum Disilicide Coatings on Molybdenum. E. A. BEIDLER, C. F. POWELL, I. E. CAMPBELL AND L. F. YNTEMA. *J. Electrochem. Soc.*, **98,** 21-5 (1951). Mo was treated in a $SiCl_4$-H_2 atmosphere from 1000 to 1800

C. The formation of MoSi and $MoSi_2$ was determined by X-ray and metallographic analysis.

Electron Microscopic Investigation of Metal Oxide Layers. G. PFEFFERKORN. *Z. Metallkunde,* **46,** 204-7 (1955); *Naturwissenschaften,* **40,** 551-2 (1953). The characteristics of needle growth on Mo during incipient oxidation at 220 and 310 C were studied with the electron microscope.

High Pressure Oxidation of Metals. Technical Report No. 6. Molybdenum in Oxygen. R. C. PETERSON AND W. M. FASSELL. University of Utah, Department of Metallurgy, Report for Department of the Army Project 599-01-004, Sept., 1954, (PB 123817). Abstracted in *U. S. Government Research Report,* **27,** (1957) Jan. 18. The temperature and pressure dependence of the oxidation of Mo in oxygen was studied in the ranges 525-700 C and 1-47.6 atmospheres.

Kinetics and Mechanism of the Oxidation of Molybdenum. M. SIMNAD AND A. SPILNERS. *Trans. Am. Inst. Mining Met Engrs.,* **203,** 1011-16 (1955). The oxidation kinetics were investigated between 500 and 770 C in oxygen at 1 atmosphere pressure. X-ray analysis was used to identify MoO_2 and MoO_3 as inner and outer oxide layers. Marker movement experiments indicated that the oxides grow almost entirely by anion diffusion.

The Properties of Oxidation-Resistant Scales Formed on Molybdenum-Base Alloys at Elevated Temperatures. M. GLEISER, W. L. LARSEN, R. SPEISER AND J. W. SPRETNAK. Symposium on Basic Effects of Environment on the Strength, Scaling, and Embrittlement of Metals at High Temperatures, ASTM Special Technical Publication No. 171, 65-88 (1955). Mo-Ni and MoCo alloys form protective molybdates at elevated temperature. A phase transformation in the molybdates results in spalling on thermal cycling. However, spalling can be suppressed by the addition of third components, which stabilize the molybdate crystal structure. X-ray diffraction and differential thermal analysis were used.

Search for Oxidation-Resistant Alloys of Molybdenum. G. W. P. RENGSTORFF. *Trans. Am. Inst. Mining Met. Engrs.,* **206,** 171-6 (1956). Mo-Ca and MoNi alloys form protective molybdates at elevated temperature. Many other Mo alloys were oxidized.

Results of Research Into the Kinetics of the Oxidation of Molybdenum and Tungsten and Into the Nature of the Resultant Oxides. K. M. GORBOUNOVA AND V. A. ARSLAMBEKOV. *J. Chim. Phys.,* **53,** 871-83 (1956). Oxygen absorption and oxidation of Mo were studied at various temperatures, pressures and surface preparations by using the weight and pressure change methods. A quantitative measure of evaporation and reduction of the oxides by hydrogen was used to determine the presence of MoO_2 and MoO_3.

PART 15—NICKEL AND NICKEL ALLOYS*

Corrosion Products

The Investigation by Electron Diffraction of the Corrosion of Metals. S. YAMAGUCHI. *Bull. Chem. Soc. Japan,* **18,** 53-91 (1943) (German); *Chem. Abs.,* **42,** 1864 (1948), March 20. Atmospheric and other corrosion tests on a wide range of metals and alloys, including Ni and Hastelloy A. Study of film structure and corrosion products formed on Ni by N, HCl and HI. Notes on a phenomenon, termed "selective oxide structure" in which the oxide of only one of the ingredients of the alloy is formed on the surface (in connection with Hastelloy A). A number of electron-diffraction photographs of corrosion and oxidation products are shown.

Attack of Various Superheated Steam Atmospheres Upon Aluminum Bronze Alloys. A. P. C. HALLOWES AND E. VOCE. Based on B.N.F.M.R.A. Report R.R.A. 527, Pt. II. *Metallurgia,* **34,** No. 201, 119-122 (1946) July. Failure of hollow truncated DTD197 (10Al-5Fe-5Ni) bronze cones used as plungers in manufacture of glass tumblers, and of Fe-Mn Bronze valve bodies controlling a superheated steam supply led to experiments with 10, 11, 12, 2, 5 Al bronzes, DTD197 bronze, 60-40 brass, 88-10-2 and 90-10 Sn- bronzes in pure steam and steam contaminated with various concentrations of SO_2 and Cl_2 at various temperatures and for varying lengths of times. Pure steam was harmless. Prolonged exposure to steam contaminated with either Cl_2 or SO_2 at elevated temperature produced attack on all but the 60-40 brass. Similar results were obtained with tap and distilled water. Nature of corrosion products is described and microstructures are shown.

Corrosion of Metals in Acid Fumes. W. FEITKNECHT. *Schweizer Chemiker-Zeitung,* **29,** 332-333 (1946) Oct. (German); *Metals Rev.,* **20,** No. 2, 18 (1947) Feb. The corrosion of Zn, Cd, Ni, and Fe in HCl and HBr vapor was investigated. Chemical natures of the corrosion products formed at different vapor pressures (concentration) determined.

Attack of Metals by Moist Vapours of Halogen Acids. W. FEITKNECHT. *Helv, Chim. Acta,* **29,** 1801-1815 (1946) Dec. 2; *Chem. Abs.,* **41,** No. 5, 1193-1194 (1947) March 10. Visual examination and X-ray diffraction studies were used to follow the course of the reaction of air containing vapors of HCl or HBr on Zn, Cd, Ni, and Fe, and to determine the rate of attack. For the initial stages of the attack, the reactivity of the surface oxide layer is the determining factor. Oxide films on Ni and Fe react only slowly or not at all with dilute acid vapors with the formation of basic salts. Attack starts at weak points in the film and spreads from these. In later stages of corrosion, electrochemical processes become predominant. Solid reaction products may be hydroxides, basic salts or normal salt. Each is discussed. A very low effectiveness (defined as the ratio of the weight of corrosion product actually formed to the theoretical) is found when the oxide film is very resistant to attack, as with stainless steel.

Long Term Metals Corrosion Investigation. U. S. Panama Canal, Special Eng. Div., First Interim Report, Jan. 1947, No. 74, 78 pp., Appendix 1, 167 pp. Significant information and data relating to major corrosion investigation of 52 different metal types, including steels, stainless steels, Cu-Ni, Monel, and Ni. Embraced are tropical fresh-water, sea water, and atmospheric exposures. Compilation includes organization and initiation of plan, exposure sites, environment and durations, design of equipment and apparatus, processing and installation of specimens, inspection of tests of metal samples and marine growths and physical properties, analysis of metals, immersion media and corrosion products and an extensive bibliography. The appendix contains photographs, tables and graphs to illustrate.

Literature Review on Corrosion of Metals and Materials by Flue Gas Condensate. H. A. PRAY, F. W. FINK AND R. S. PEOPLES. Amer. Gas Assn. Report No. 1, Project DGR-4CH, Feb. 1947, 29 pp. Review of corrosion problems, and sources of fuel gases and typical compositions are given. Origin and form of S in fuel gases, removal of organic S compounds, and the properties of the products of combustion of fuels are discussed. Corrosive factors involved and corrosion products, and laboratory tests on corrosion of metals and materials are described. Metals studied include Ni and stainless steels. 55 references.

Condensate Return Line Corrosion Control. H. N. POTTER; **Some Notes on Corrosion in Condensate Return Lines.** F. L. LAQUE. *Modern Power & Eng'g,* **41,** No. 5, 47-48, 108 (1947) May; No. 12, 47-48 (1947) Dec. Original article by Mr. Potter describes use of inhibitors to mitigate corrosion in condensate return lines. Mr. LaQue's notes, provided upon invitation of editor as sequel, present corrosion data from tests at Bayonne by Mr. Copson, on the behavior of various metals and alloys in distilled water, at various temperatures, with water saturated with various mixtures of CO_2 and air. Conclusions were that Sn, Inconel and 18-8 stainless were the best materials, in that order. Conclusions were supported by service data from L. F. Collins. Mr. LaQue points out the protective nature of the corrosion products, the fact that complete deaeration of boiler water and steam would not completely prevent corrosion if CO_2 were present, but that it is more important for steam to be free of air than of CO_2, and that effects of temperature of condensate varies with ratio of CO_2 to air in saturated gas. He points out in conclusion that investigations point the way to corrosion control in condensate handling systems by: (1) elimination or reduction of corrosive

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agents by feed water treatment and venting of noncondensible gases; (2) use of inhibitors; (3) design and construction of systems to exclude air as far as may be practical; and (4) use in critical services of metals and alloys shown to be corrosion resistant under the most unfavorable conditions.

The Breakdown of Oxide Films in Acid Vapors. W. FEITKNECHT. Paper before Pittsburgh Internat. Conf. on Surface Reactions, June 7-11, 1948; *Proc. Pittsburgh Internat. Conf. on Surface Reactions,* 1948, 212-221. Discussion of facts about corrosion of Fe, Zn, Cd, Ni and Cu in air containing HCl vapor. Three kinds of breakdown of primary oxide film are distinguished; on Ni and Fe primary oxide film breaks down only at isolated points; on Zn and Cd whole film breaks down and grows thicker; and on Cu a film of cuprous oxide grows and interference colors are seen. Initial breakdown is thought to occur by an electrochemical mechanism in each case, and subsequent course of reaction is determined by properties of possible solid corrosion products. Course of corrosion was followed by determination of changes in weight, and electron microscope was used to follow initial stages. Corrosion products were identified by microscopic and X-ray examinations, and mechanism of corrosion for each metal is discussed in detail.

Effect of Non-Condensable Gases on Corrosion of Nickel in Steam Condensate. W. A. WESLEY AND H. R. COPSON. *J. Electrochem. Soc.,* **95,** No. 5, 226-241 (1949) May. Discussion includes field observations, apparatus, standard procedure, effects of temperature, composition of non-condensable gases, time, and pressure, passivity of Ni in steam condensate, corrosion rate of Ni in presence of Fe, effect of purity of condensate, corrosion product, corrosion of Fe in steam condensate, corrosion of Inconel, corrosion of other metals and preventing condensate corrosion of Ni tubes. Corrosive conditions to which chemical evaporator tubes may be exposed on steam side were simulated in corrosion tests run in hot water saturated with mixtures of CO_2 and air under pressure. These showed that appreciable corrosion of Ni can occur in a critical range of composition of non-condensable gases, namely from about 55-90 percent CO_2 by volume. In presence of Fe corrosion products, this range is broadened to include 40-90 percent CO_2. Outside critical range, Ni owes its good resistance to development of a passive film. Corrosion of Ni is prevented by removing air or CO_2 from steam until their ratio is no longer critical. Inconel, Sn, and stainless steel were resistant to attack under all experimental conditions. Zn and some Cu alloys were also tested. Five tables include one on corrosion of various metals. Among graphs are those illustrating effects of temperature, composition of non-condensable gases at various temperatures, and time on corrosion rate of Ni in steam condensate.

Surface Problems of the Chemical Behavior (Corrosion) of Metallic Materials. W. GUERTIER. *Metalloberflache,* **3,** Sect. A, No. 7, 133-141, (1949); *J. Inst. Metals & Metall. Abs.,* **18,** Pt. 2, 116 (1950) Oct. Discussion of the relationship between chemical attack and the crystal structure of the material. Where corrosion results in removal of material then, in the case of single crystals, the resistance to attack decreases in the order; face-centered cubic hexagonal body-centered cubic structure. Differences in the rate of reaction also occur within the individual types of crystal, as a result of the differing orientations of the constituent atoms at the various corners, edges, and plane surfaces. Solid solutions behave similarly to single crystals. Heterogeneity increases corrosion, especially at corners and edges. When the behavior of polycrystalline aggregates is considered the relative orientations of the individual crystals must be taken into account. Where chemical attack results in the formation of corrosion products on the surface of the material, the progress of the reaction is determined by the nature and properties of such films. The corrosion and corrosion-resistance of Fe, Co, Ni, Al, and Mg and their alloys, and stainless steels, are discussed in relation to the above mentioned factors.

Fracture of Oxide Films on Metal Surfaces in Acid Vapor and the Mechanism of Atmospheric Corrosion. W. FEITKNECHT. *Chimia,* **6,** 3-13 (1952); *Brit. Abs.,* B I, May 1952, 676. The investigation of the corrosion of Zn, Cd, Ni, Fe, and Cu by HCl vapor is carried out. Samples are suspended in an atmosphere of known HCl content, saturated with water vapor. The products of corrosion are investigated by X-ray analysis. Surface changes in the metal during corrosion are investigated, at first microscopically, and later by means of the electron-microscope. Three types of oxide film fracture can be differentiated. Corrosion of metals in moist acid vapor is an electrochemical process. All the important facts relating to the chemical and thermochemical properties of the corrosion products of Zn, Fe, Ni, and Cu are gathered together and the mechanism of corrosion in each case is discussed.

Porosity of Electrodeposited metals. XI. Analysis of Corrosion Products as a Method of Measuring the Change of Porosity in Corrosion. N. THON L. YANG AND S. YANG. A.E.S. Res. Proj., No. 6 *Plating,* **40,** No. 9, 1011-1015 (1953) Sept. Method by which the progress of corrosion could be determined, as a function of time exposure, was devised. An analytical procedure was devised to collect all corrosion products and at the same time not corrode any of the base metal. Examples are given of the application of this method to various corrosive exposures, including hot-water immersion with and without access of air, intermittent salt immersion, and exposure to a corrosive gaseous atmosphere. Samples of Ni coatings were tested. Tables.

Oxidation of Iron-Molybdenum and Nickel-Molybdenum Alloys. S. S. BRENNER. *J. Electrochem. Soc.,* **102,** No. 1, 7-15 (1955) Jan. Mo significantly alters the high temperature oxide structure on Fe and Ni. Binary alloys of Fe and Mo (up to 12.5 atomic percent Mo) and Ni and Mo (up to 19.7 atomic percent Mo) do not exhibit catastrophic oxidation up to 1000 C. In both systems MoO_2 and the Fe or Ni molybdate are formed in addition to the oxides. Mo decreases oxidation rate of Fe by preventing formation of cation defficient FeO normally formed above 570 C. Oxidation rate of Ni is affected only to a small extent. Up to 13.1 atomic percent Mo the rate is increased by formation of additional vacancies in the NiO lattice due to dissolved Mo atoms; beyond 13.1 percent it decreases due to densification of MoO_2 subscale. Mechanism of oxide growth is discussed. Graphs, tables, 13 references.

Catastrophic Oxidation of Some Molybdenum-Containing Alloys. S. S. BRENNER. *J. Electrochem. Soc.,* **102,** No. 1, 16-21 (1955) Jan. Catastrophic oxidation occurs in certain concentration regions when Ni or Cr is added to binary Fe-Mo alloys. Cr induces this rapid oxidation more readily than Ni. After initial oxidation, metal surface becomes enriched in Mo, and the oxide on the alloy cracks, allowing formation of liquid MoO_3 along alloy oxide interface. Catastrophic oxidation does not occur in alloys with large amount of Ni; formation of MoO_3 is prevented as in Ni-rich Ni-Mo alloys. Table presents data on loss of metal by oxidation during 2 hr at 1000 C for Mo-Fe-Ni, Mo-Cr-Fe, and Ni-Mo-Fe-Cr of various compositions. Graphs, illustrations, 6 references.

Rate of Flow as Factor of Special Importance in Corrosion by Sulphuric Acid. H. W. VAN DER HOEVEN. *Werkstoffe und Korrosion,* **6,** 57-62; disc. 62, (1955) Feb. Short review of available data on corrosion by H_2SO_4, from which it is concluded that laboratory data do not permit critical selection of materials for practical applications. Description is given of new laboratory test method developed to permit measurement of corrosion under different conditions of flow. Investigation was made of effect of accumulation of corrosion products in the attacking solution. Data are presented of measurements on C steel and Ni-Al bronze.

Progress Report of Task Group T-4F-1 on Water Meter Corrosion. R. W. HENKE. *Corrosion,* **12,** No. 1, 77-80 (1956) Jan. Susceptibility of water meters to corrosion is discussed especially the manner in which corrosion affects operation of individual parts. Case studies are given with accompanying illustrations. Photomicrograph shows a corrosion deposit consisting of Cu chloride, ferrous oxide, ferric oxide and a complex of Ni carbonate and hydroxide. Discussion includes economic aspects of water meter corrosion, nature of the corrosion problem and factors contributing to corrosion.

High Temperature—High Pressure Corrosion Of Zirconium in Hydrochloric Acid*

By W. E. KUHN

Abstract

The corrosion of zirconium in HCl was studied at 150-250 C in aqueous solutions containing 5 to 37 percent by weight of HCl. Studies of the dependence of the corrosion rate on temperature and time were conducted, and the experimental data correlated with studies of the corrosion products, surface films, and the metallurgical structure and chemical composition of the substrate. The kinetics are also given.

Hydrogen was found to play an important role in the corrosion mechanism as a surface coating of zirconium monohydride and dissolved in the zirconium lattice.

Initially, an accelerating corrosion rate was found with time, followed by a transition to a decreasing corrosion rate. It was concluded that the corrosion mechanism was controlled by the initial reaction up to the transition point, beyond which it is suggested that the reaction is either diffusion controlled and/or influenced by hydrogen adsorbed at the surface.

Intergranular corrosion of alpha-annealed zirconium and attack at both the grain boundaries and between the alpha needles of beta-treated furnace cooled zirconium were observed at the higher temperatures. The beta-treated furnace cooled zirconium was more resistant to attack by HCl than the alpha-annealed zirconium.

Much improved corrosion resistance to HCl may be expected with higher purity metal. In this respect the zirconium used in this study does not represent the higher quality metal being produced today by advanced processing, melting and fabrication techniques. 6.3.20

Introduction

THERE ARE indications that zirconium possesses markedly better corrosion resistance to hydrochloric acid than is indicated in the literature, and that the presence of minor impurities prevents realization of the metal's full potential.

An understanding of the mechanism of corrosion of zirconium in hydrochloric acid is prerequisite to effective use of this metal in corrosion applications, to any major improvement in its corrosion resistance, and to planning of future studies. Since corrosion is a surface phenomenon, an attempt was made in this study to correlate the experimental data with studies of the corrosion products, surface films, the metallurgical structure and chemical composition of the substrate.

It is hoped that these studies, by providing a clearer insight and understanding of the corrosion behavior of zirconium, will lead to chemical compositions and heat treatment procedures that will enhance the already outstanding corrosion properties of zirconium.

About the Author



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Test Procedure and Materials

Test Procedure

Standard coupons ½ inch by 2 inches by 0.045 inches are sealed in heavy-walled borosilicate tubes filled one-third with the corrosive fluid in sufficient amount to fully immerse the specimens. The procedure employed is similar to that described in references 1 to 6.

The surface of the specimens was finished with 120 grit silicon carbide abrasive cloth to remove about 0.001 to 0.003 inches of the surface metal and expose metal representative of the internal structure of the metal. Specimens were cleaned thoroughly with acetone and dried at 70 C in air prior to weighing and sealing into the borosilicate tubes. After sealing, the specimens were placed in iron pipe protective tubes, preheated to minimize the heating time. The test duration was taken as the total elapsed time from the time the temperature reached 95 percent of the test temperatures. The acid concentration was determined after each test. Tests were generally conducted in duplicate and prediction equations were established by the method of least squares with 95 percent confidence limits through Univac I programming.

Materials

Commercial hydrochloric acid of standard chemical purity was employed throughout these tests. The zirconium employed was hot rolled strip prepared from an argon arc melted zirconium ingot by a steel fabricating company. The sheet was sandblasted, caustic pickled to remove the oxidized surface layer, and level stretched for straightness.

The chemical composition is given in Table 1. The metallurgical structure is shown in Figure 1 to be equiaxed alpha-annealed and to have an average grain size of 8.5 or 0.0008 inch, in accordance with ASTM Designation on E91-51T for nonferrous metals other than copper and brass. The average diamond pyramid hardness was 162 in the longitudinal direction and 154 in the transverse direction, using a 5 kg load.

Advances in processing, melting, and fabricating have resulted in improved grades of commercial zirconium, hence the corrosion data reported herein do not represent the higher purity grades available today.

Test Results

Dependence of Corrosion Rate on Temperature and Hydrochloric Acid Concentration

A logarithmic graph of the corrosion weight loss versus the reciprocal of the absolute temperature at each level of acid concentration is given in Figure 2. The straight lines indicate that the data are satisfactorily represented by an Arrhenius type relationship.

$$\text{Log}_{10}\text{K} = \frac{\text{E}_{\text{exp.}}}{2.303\ \text{RT}} + \text{Log}_{10}\text{A} \quad (1)$$

Where K is the velocity constant, $\text{E}_{\text{exp.}}$ is an experimental energy of activation or temperature coefficient of activation, R is the gas constant, T the absolute temperature, and A a constant called the frequency factor.

The corrosion weight losses at 225 C and 250 C at each acid concentration are significantly lower than the values predicted by extrapolation of the straight line relationship. An explanation for this is given in section entitled "Discussion of Test Results."

The temperature coefficients for the log weight loss versus the reciprocal of the absolute temperature have been calculated for each concentration by the method of least squares and are tabulated in Table 2. The average temperature coefficient for the five acid concentrations is 32,360 ± 1600 calories per mole. Values for the frequency factor A in terms of milligrams per square decimeter per 48 hours were recalculated using a value E = 32,360 calories per mole and have been plotted in Figure 3. Thus using the relation:

$$\text{Log}_{10}\text{R} = \frac{-32{,}360}{2.303\ \text{RT}} + \text{Log}_{10}\text{A} \quad (2)$$

and substituting values for Log A from Figure 3, the corrosion rate R at any concentration between 15 and 37 weight percent hydrochloric acid and temperatures between 100 and 200 C may be estimated for a test cell 33 percent filled with acid solution.

Dependence of Corrosion Rate on Time and Temperature

Figure 4 is a log-log plot of the weight loss versus time at different tempera-

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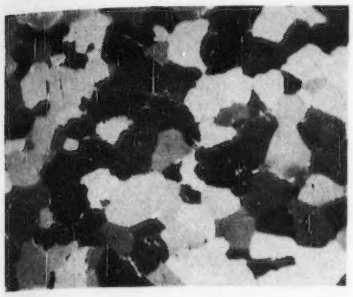


Figure 1—Photomicrograph showing the structure of the zirconium used in the corrosion tests in the as received condition. Structure is that of alpha-annealed sheet after level stretching. Chemical polished with HNO_3-H_2O-HF solution. Polarized light. Approx. 250X.

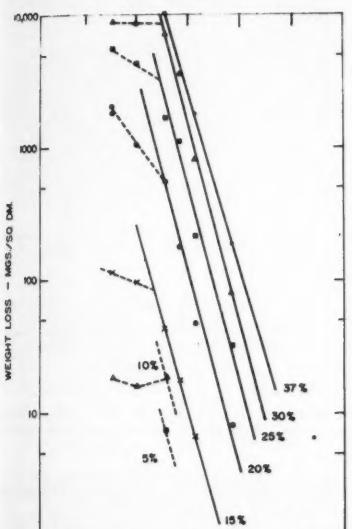


Figure 2—Dependence of the corrosion weight loss of zirconium in 48 hours on temperature and hydrochloric acid concentration.

tures. The straight line portions of each curve may be represented by an empirical equation of the form

$$W = Ct^n \qquad (3)$$

where W is the unit weight loss after time t, n is the slope of the curve, and C a rate constant.

Values for n and the rate constant C have been determined by the method of least squares and are given in Table 3.

In Figure 5 the initial slopes of the time versus corrosion weight loss curves have been plotted against the reciprocal of the absolute temperature. The values for the slopes were determined from linear plots of the data represented in Figure 4 at each temperature by drawing the tangent to each curve at time zero. A typical Arrhenius plot is obtained with a temperature coefficient of 31,319 ± 2,002 calories per mole and a frequency factor $\log_{10}A = 15.15 \pm 0.950$ where A is in milligrams per sq dm per 48 hours.

A summary of the results of X-ray and optical examination of the surface of the specimens corroded at 200 C in 20

TABLE 1—Chemical Purity of Commercial Grade Zirconium Used in Corrosion Studies

SPECTROGRAPHIC ANALYSIS (Parts per Million)

Hf	Al	Cr	Fe	Mg	Mn	Ni	Pb	Ti	V	Si	Cu	Co
2%	250	320	1140	20	300	<20	50	40	<20	90	21	<20

Hydrogen (2) * 57.5 Outside laboratory determination (2) 63.
Oxygen (2) 750 parts per million.
Nitrogen (1) 65 parts per million.
Carbon (2) 50 parts per million.
Chlorine (2) less than 20 parts per million.
* Number in Parentheses Represent Number of Determinations.

TABLE 2—Summary of Temperature Coefficients and Frequency Factors Calculated for Different HCl Acid Concentrations in Test Cells ⅓ Filled With Acid Solution

HCl Concentration Weight Percent	Temperature Coefficient of Activation E_{exp} (calories per mole)	Frequency Factor Log 10 A	
		Experimental Values	Calculated on Basis of E = 32,360 Cals./mole
15	−29,771 ± 4390	15.422 ± 2.143	16.673
20	−33,635 ± 3394	18.273 ± 1.652	17.652
25	−33,015 ± 6399	18.597 ± 3.119	18.255
30	−36,664 ± 3501	20.910 ± 1.707	18.831
37	−31,708 ± 2235	18.736 ± 1.098	19.038
Overall or pooled value	−32,359 ± 1606	See Figure 3	.....

percent hydrochloric acid is given in Table 4. At 200 C in 20 percent hydrochloric acid, black delta zirconium monohydride[1] and monoclinic zirconium oxide were identified in the corrosion products formed between 6 and 312 hours. Pitting attack became evident after about 72 hours and at 312 hours had the appearance shown in Figure 6.

Dependence of Corrosion on Structure and Surface

The results of X-ray and metallographic studies of the surface of specimens exposed to 20 percent HCl solution at various temperatures are given in Table 5. Black monohydride and monoclinic zirconium oxide were identified in the surface films at all temperatures.

Monoclinic zirconium dioxide has been observed and identified as a submicron white powder which oozes from the black film while rinsing the specimens after testing. After corrosion tests in which a large amount of zirconium was dissolved, a voluminous precipitate was obtained which on gentle drying was identified as zirconium oxychloride.

The effect of corrosion on the surface is shown in Figure 7 after exposure to 20 percent HCl at temperatures between 150 and 250 C inclusive. A dark phase, appearing white under polarized light in Figure 8, follows the grain boundaries. Analysis of the surface by X-ray diffractometer after erasing the outer black film shown in Figure 9 reveals residual zirconium monohydride and monoclinic zirconium oxide. In Table 5 the intensity of the characteristic lines for monoclinic zirconium dioxide increased sharply with temperature, whereas the intensity of the hydride phase remained relatively unchanged.

Comparison with the microstructure of uncorroded zirconium revealed no perceptible increase in the number of hydride needles in the structure adjacent to the corroded surface nor in the interior of the specimens.

Samples of the specimens represented in Table 5 and Figure 7 were abraded to remove 0.004 inch from all surfaces well below the thickest corrosion-affected layer. These specimens were analyzed for hydrogen with the results given in Table 6.

Up to 225 C, hydrogen pick-up is relatively minor but increases sharply at 250 C. The sudden increase in hydrogen concentration at 250 C coincides with the temperature region where hydrogen begins to diffuse rapidly into the zirconium lattice.[12]

[1] The black films produced at the higher temperatures were too thin to give more than a single strong reflection from the (111) plane of the face centered cubic crystal structure against the strong background radiation. It was assumed that the reflection from the (111) plane having a d spacing of 2.76 Å was the same as that from thicker black films formed on the surface of electrolytically polished specimens exposed to boiling 20 percent hydrochloride acid identified in earlier work and having a lattice constant $a_0 = 4.7738 \pm .0018$ Å.[7]

The X-ray diffraction pattern for this same specimen was redetermined using NaCl for calibration with the results summarized in the table below. The lattice constant a_0 was calculated to give a value of 4.7788 Å using corrected d values for the (311) and (222) planes. The reflections from higher Miller indices planes were too weak to be identified and reported in table below.

Interplanar Spacings (d), Measured Intensities (I − I_o), and Miller Indices (hkl) of the Face-Centered Cubic Crystal Structure of the Zirconium-Hydrogen Phase Present in the Corrosion Product Film.

d (Å) corrected	(I - I_o) cms. over background	hkl.
2.762	15.0	111
2.399	6.0	200
1.6911	3.5	220
*1.4426	2.8	311
*1.3810	1.2	222

* These values were used for unit cell edge calculations giving $a_0 = 4.7788$Å.

A distorted cubic or tetragonal phase having a lattice constant of nearly the same value has been reported to be in equilibrium with the metal in the analogous hafnium-hydrogen system.[8] The tetragonal phase is characterized by line splitting and groups of lines where single reflections in the cubic phase occur.[8, 9] Since these were absent in the cubic phase identified in the corrosion product, it is concluded that this is Hagg's delta face centered cubic modification.[10, 11] Since only the reflection from the (111) plane could be obtained from the films produced at the higher temperatures, it is not known with certainty whether the hydride formed is the face centered cubic or the deformed cubic modification, although it is believed to be the former.

Monoclinic zirconium dioxide was positively identified in the surface corrosion product by a clearly defined X-ray diffraction pattern.

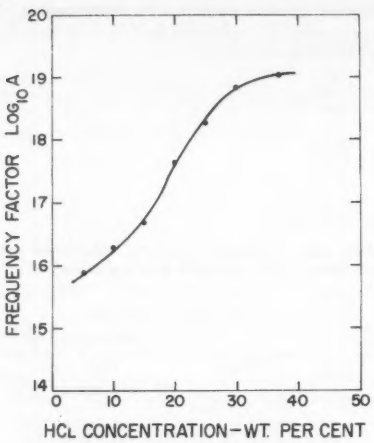


Figure 3—Dependence of frequency factor A on the concentration of hydrochloric acid.

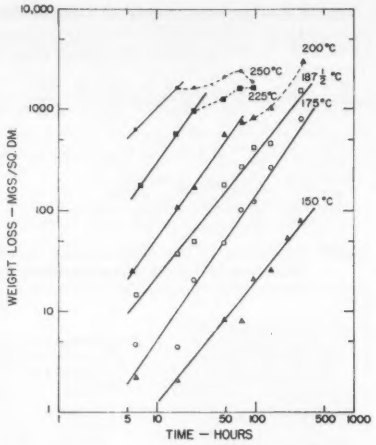


Figure 4—Dependence of the corrosion weight loss of zirconium on time and temperature in 20 percent hydrochloric acid solution.

TABLE 3—Tabulation of Values of the Surface Activity Factor S and the Rate Constant C and Exponent n in the Relation $W = Ct^n$ at Various Temperatures in 20 Percent HCl

Temp. Degrees C	Rate Constant C, mgs/sq.dm./t^n	n	Initial Rate Constant k mgs/sq.dm./hr.	Relative Surface Activity Factor	
				After 48 Hours	At Time of Transition
150	.08	1.20	0.08	2.56	.....
175	.20	1.43	0.70	2.10	.....
187.5	1.37	1.21	1.95	1.92	.....
200	3.51	1.28	4.0	3.28	(65 Hrs) 3.62
225	12.39	1.37	19.3	3.68	(24 Hrs) 2.85
250	108	0.98	106	1.0 (1 Hr)	(16 Hrs) 0.95

TABLE 4—Summary of X-Ray Data and Visual Observation of Specimens Corroded At 200 C in 20 Percent Hydrochloric Acid

Duration of Test (Hrs)	Relative Intensities (I-I_0) Centimeters Over Background				Description of Corroded Surface
	Delta ZrH	Zr	Mono Clinic ZrO_2	Cubic ZrO_2	
5.5	2.2	9.8	1.8	1.2	Very thin black film, greenish tinge, no pitting, original scratches present.
16	1.9	10.7	1.7	....	Black film, no pitting, original scratches present.
24	2.0	7.8	1.4	....	Black film, no pitting, original scratches present.
72	1.8	8.1	3.2	1.6	Very black film, uniform distribution of shallow pits.
96	1.4	6.3	2.8	....	Very black film, uniform distribution of shallow pits.
312	2.15	....	5.8	....	Gray black film, uniformly but deeply pitted.

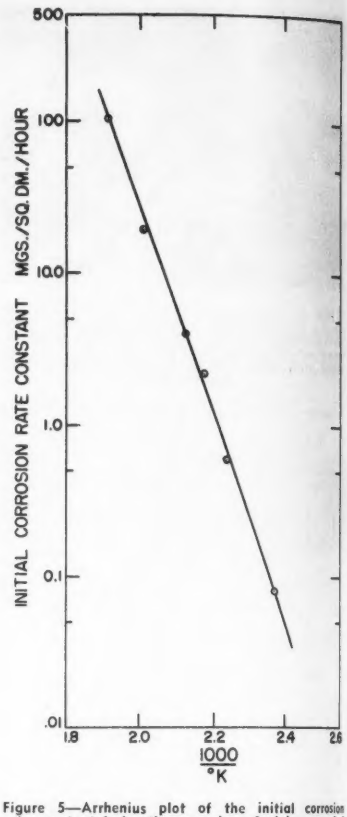


Figure 5—Arrhenius plot of the initial corrosion rate constant k for the corrosion of alpha-annealed commercial zirconium in 20 percent HCl.

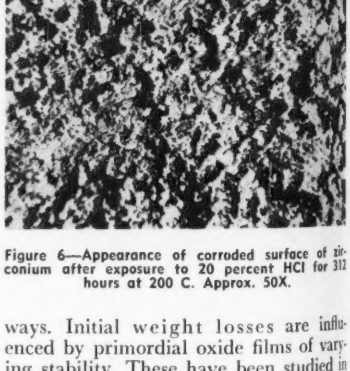


Figure 6—Appearance of corroded surface of zirconium after exposure to 20 percent HCl for 312 hours at 200 C. Approx. 50X.

Effect of Vacuum Annealing, Surface Oxidation, and Hydriding on Corrosion of Zirconium

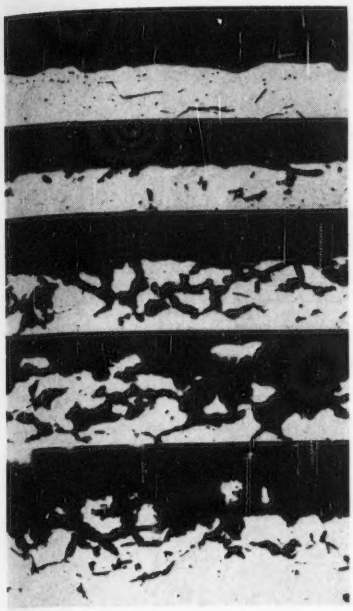
Vacuum Annealing. The as-received zirconium was beta-annealed within a zirconium enclosure at 1150 C for 3 hours in a vacuum of 0.02 micron. The leak rate of the system during the experiment was about 0.2 microns per minute. The specimens were furnace cooled to room temperature, examined, and found free of any visible film. The annealed structure, shown in Figure 10, reveals a precipitate phase concentrated between the alpha needles.

Arrhenius plots of both the beta-treated furnace-cooled and the as-received alpha-annealed zirconium are given in Figure 11. Table 7 summarizes the results of visual examination of the beta-annealed zirconium. The weight losses of the beta-annealed specimens are significantly lower than the as-received alpha-annealed zirconium, although the temperature coefficient of 32,800 calories per mole remains in close agreement with that of the latter. The value of the frequency factor was found to be $\log_{10}A = 17.480 \pm 1.028$.

Both intergranular corrosion and boundary attack between the alpha plates are revealed in Figures 12 and 13. Impurities concentrated at grain boundaries and between the Widmanstätten lamellae are believed to cause this type of attack.

Oxide Films. Surface phenomena influence the corrosion weight loss in several ways. Initial weight losses are influenced by primordial oxide films of varying stability. These have been studied in both hot water and acid solutions and are associated with a transition from a very slow to a markedly higher corrosion rate coincident with loss of oxide film by flaking.[7,13] The most tenacious and impermeable of these films are those formed on vacuum annealed zirconium.[7,14,15] Air-formed films on abraded specimens were found to be much less resistant to breakdown than those on vacuum annealed specimens.

Polishing the surface chemically or by abrasion prior to beta-annealing had no apparent effect on the corrosion rate of annealed zirconium. However, storage in the laboratory for about 19 months, fol-



Temperature of Test, Degrees C	Approximate Depth of Penetration, Mm.
150	nil
175	0.022
187.5	0.037
200	0.052
250	0.061 (24 hr duration)

Figure 7—Surface attack by 20 percent HCl after 48 hours' exposure at the temperatures indicated. Tests were conducted in heavy walled borosilicate sealed tubes filled one-third full with acid solution. Original magnification, 540X.

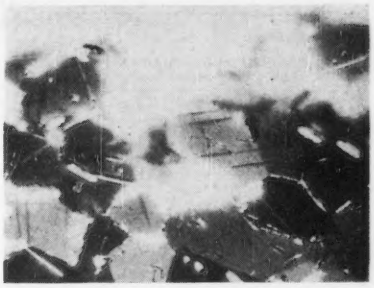


Figure 8—Intergranular corrosion of equiaxed grain structure of alpha-annealed zirconium after 48 hours exposure to 20 percent HCl at 200 C. Bright rods are hydride needles, large diffuse white areas are corroded regions. Polarized light. Approx. 500X.



Figure 9—Photomicrograph showing the appearance of zirconium after exposure to 20 percent HCl at 200 C for 48 hours. Direct bright field illumination. Approx. 50X.

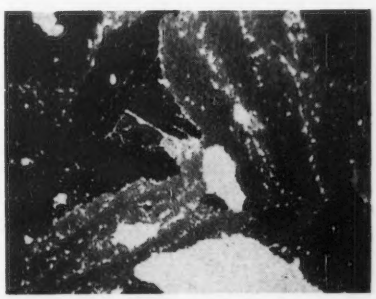


Figure 10—Photomicrograph of structure of beta-treated zirconium after vacuum annealing at 1150 C for three hours. Specimen was chemically polished in 45 cc H_2O, 45 cc HNO_3, 8 cc HF. Polarized light. Approx. 100X.

lowed by testing at 187.5 C and 175 C gave substantially lower corrosion rates, as shown in Figure 11. Although there was no evident change from the silvery white metallic surface of the specimens before exposure to the acid, a highly protective passive had evidently formed while the specimens were in storage. Removal of this film in HNO_3-HF aqueous solution resulted in a return to values which fitted the Arrhenius plot.

Specimens which had been tested shortly after vacuum annealing and after chemical polishing, exhibited the black and metallic white faceted grain structure typical of hydride formation on vacuum annealed zirconium. The untreated 19 month old films displayed the original metallic lustre, but were slightly tarnished a light yellow after exposure at 175C and a slightly deeper yellow at 187.5 C. This observation suggests that the thickness of the primordial, tenacious, impervious passive film is less than that of yellow oxide films, estimated to be about 150 Å thick.[16] Also, whereas Gulbransen found no permeation of the film of chemically polished specimens by hydrogen, this study found that the film formed by chemical polishing was not resistant to hydrochloric acid. Electron diffraction studies have not been successful in revealing the nature of the initial film, but X-ray diffraction has revealed the thicker films to be monoclinic ZrO_2.[7,15]

Zirconium Hydride. Zirconium, hydrided to produce ZrH_2 of near stoichiometric proportions, was subjected to corrosion in 20 percent HCl. Although hydriding caused the specimens to be severely distorted and cracked, they possessed sufficient strength for test purposes. The Arrhenius plot in Figure 14 for the hydrided specimens has a temperature coefficient of 10,150 calories per mole and a frequency factor of $\text{Log}_{10}A = 6.962$. It is to be noted that the Arrhenius plots of hydrided and hydrogen-free metal intersect at approximately 180 C. Above this temperature the corrosion rate of the hydride is less, and below greater, than the hydrogen-free metal.

Further evidence of the passivity of the hydride is the sharp edges of cracks in the surface of the specimens as shown in Figure 15. The interior surfaces of cracks exposed to the acid displayed a dark tarnish but were not otherwise affected by the acid. The surface area exposed to the acid was not corrected for the additional area contributed by the cracks; therefore, the actual value of the frequecy factor would be somewhat less than that reported above.

Influence of Pressure on Corrosion Rate

The HCl-H_2O system, being bivariant, will develop a definite pressure at a given temperature and composition of the solution. At constant temperature, a change in the percentage of acid solution in a test cell will affect a change in the composition of the liquid and vapor phases within the cell, and hence the corrosion rate.

The effect of acid concentration at different temperatures on the corrosion rate is shown in Figure 16 for a cell one-third filled with acid solution. It is seen that a small change in acid concentration produces a large change in the corrosion rate.

The fact that the slopes of the log-log plots are nearly parallel suggests that the mechanism is the same over the greater part of the temperatures and concentrations studied.

The change in solution concentration with temperature depends on the pressure-solubility relationship between HCl and water. Since this relationship has not been reported for temperatures above 100 C, the pressures and solution concentrations in the test cells are unknown. To obtain some idea of the sensitivity of the corrosion rate to pressure induced changes in acid concentration, a series of corrosion tests were conducted in 20 percent HCl between 150 and 200 C to determine the corrosion weight loss at various solution levels. A log-log plot of the percentages of the test cell filled with acid and the weight loss at different temperatures after 48 hours is given in Figure 17.

These curves show that the hydrochloric acid concentration of the solution increases with increasing solution level in the cell, attaining the nominal acid concentration (20 percent) when the cell is 100 percent filled. The increasing slope of the weight loss versus solution level

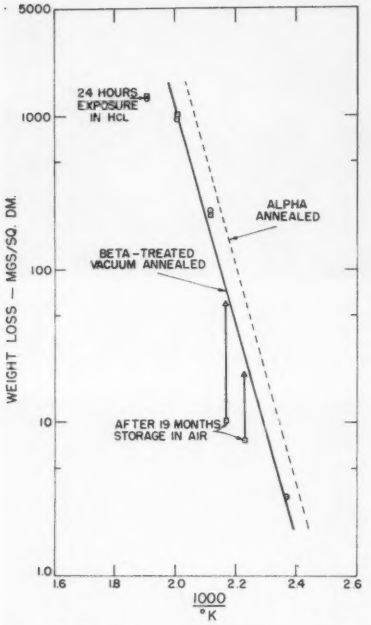


Figure 11—Dependence of the corrosion weight loss of beta-treated vacuum annealed zirconium on temperature for an exposure of 48 hours in 20 percent HCl compared with alpha-treated structure of the "as received" zirconium. Code to symbols is as follows: Circles—chemical polished with HNO_3-H_2O-HF polishing solution before annealing. Tested after 5 to 24 days' exposure to air; Squares—surface finished with 120 grit silicon carbide abrasive cloth before annealing. Except where noted, tested after 5 to 24 days' exposure to air; Triangles—surface finished with 120 grit silicon carbide abrasive cloth before annealing and chemical polished with HNO_3-H_2O-HF polishing solution after annealing.

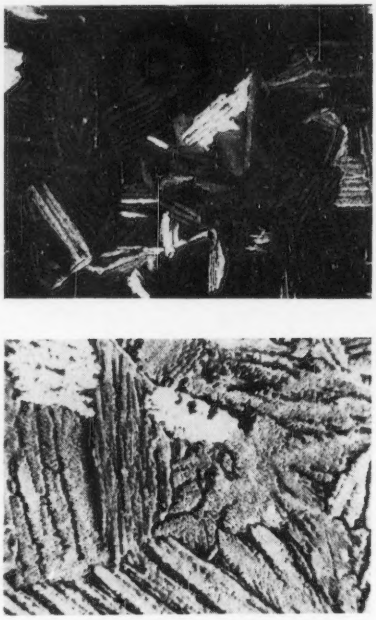


Figure 12—Photomicrographs showing the appearance of the corroded surface of beta-treated vacuum annealed zirconium after exposure to 20 percent HCl at 250 C for 24 hours. Direct oblique lighting. Top photomicrograph is at a magnification of approximately 13X; bottom photomicrograph approximately 50X.



Figure 13—Photomicrograph of cross section of surface shown in Figure 12 showing interlamellar corrosion between Widmanstatten plates where impurity phases have precipitated. Polarized light. Approx. 100X.

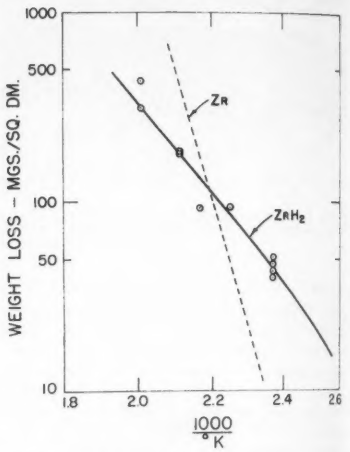


Figure 14—Dependence of the corrosion weight loss of zirconium hydride on temperature compared with zirconium metal for an exposure of 48 hours in 20 percent hydrochloric acid.

curves with increasing temperature, indicate that for a given solution level, the solubility of hydrochloric acid decreases with increasing temperature.

An Arrhenius plot of the extrapolated weight losses from Figure 17 for a test cell 100 percent filled gave a temperature coefficient of 36,436 ± 15,728 calories per mole and a frequency factor of $\log_{10} A = 19.90 \pm 7.44$. These represent values corrected for pressure effects induced by temperature and hydrogen evolution. The large confidence limits are partly a result of having only four points available to estimate the slope and intercept of the Arrhenius relationship but chiefly are the consequence of having to extrapolate from other data to obtain these points. The best estimate of the value of the activation energy is the mean, 36,436 calories per mole.

Effect of Molecular Hydrogen, Oxygen, Nitrogen, and Argon in The Vapor Phase on Corrosion

Molecular gases were introduced into the evacuated space above the liquid phase in the test cells by first evacuating the borosilicate tubes, degassing the acid solution, and then back-filling with tank hydrogen, oxygen, nitrogen, and argon. The results of corrosion tests at 200 C in 20 percent HCl for 48 hours are given in Table 8. The results show that the amounts of these gases present have no aparent effect on the corrosion rate of zirconium at 200 C.

TABLE 5—Summary of X-Ray Data and Visual Observations of Specimens Corroded At Various Temperatures in 20 Percent HCl for 48 Hours

Temp. Degrees C	Relative Intensities (I-I_o) Centimeters Over Background Radiation				Description of Corroded Surface
	Delta ZrH	Zr	Mono Clinic ZrO_2	Cubic ZrO_2	
150	1.6	6.2	1.6	....	Thin, black lustrous surface film, few shallow pits.
187.5	1.7	10.0	2.2	1.3	Black film, no pitting evident, original scratch lines still present.
200	2.7	9.6	2.9	(1.9)	Black film, pitting observed, original scratch lines evident.
225	2.0	8.9	3.6	....	Black film, uniform distribution of shallow pits, original scratch lines still evident.
250*	1.6	8.1	5.7	....	Thick greyish black porous coating having rough pitted appearance.
250**	2.15	....	7.7	....	Thick greyish black coating having rough pitted appearance. Coating very porous and easily removed by light scraping. Appears light grey under low magnification.

* 24 hours exposure.
** 48 hours exposure.

Discussion of Test Results

Surface Activity Factor

The fact that the temperature coefficient of the initial reaction and the overall reaction, 31,800 and 32,360 calories per mole respectively, are in close agreement suggests that the corrosion mechanism is controlled by the initial reaction at temperatures between 150 C and 250 C inclusive for hydrochloric acid concentra-

TABLE 6—Effect of Temperature on Hydrogen Absorption in 20 Percent Hydrochloric Acid

Temp. Degrees C	Exposure Time (Hours)	Hydrogen Analysis (Parts Per Million)	Increase in Hydrogen Absorption Over Original Hydrogen Content (Parts Per Million)
..	..	(2*) 63**	0
150	48	(2) 70.5	7.5
175	48	(2) 78.5	15.5
187.5	48	(1) 78.0	15.0
200	48	(2) 88.5	25.5
225	48	(1) 93	30.0
250	24	(1) 116	53.0

* Number of determinations
** As received material

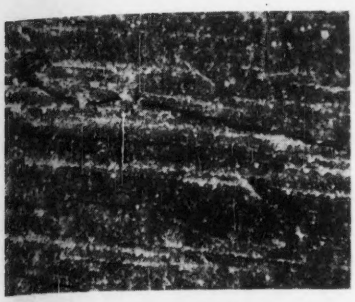


Figure 15—Photomicrograph showing the appearance of the surface of zirconium hydride after exposure to 20 percent HCl for 48 hours at 200 C. Note the sharp edges of the crack. Direct illumination. Approx. 50X.

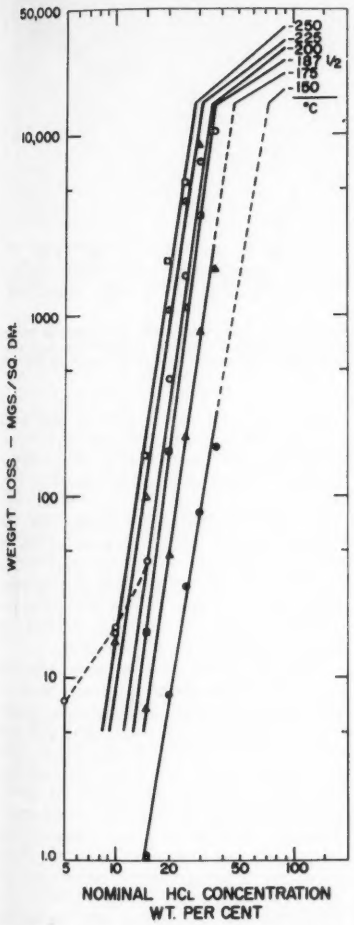


Figure 16—Dependence of corrosion weight loss on nominal hydrochloric acid concentration at different temperatures for an exposure of 48 hours.

tions of 15 to 37 percent. The mechanism assumed to explain the slow step is given by the expression:

$$\frac{dW}{dt} = kS \qquad (4)$$

where $\frac{dW}{dt}$ is the instantaneous rate of dissolution at time t, k is the initial corrosion rate, and S a complex surface sensitive factor. The factor S is influenced by one or more of several factors including acid concentration, adsorbed gases and ions, amounts and distribution of impurities in either acid solution or metal, metallurgical structure, etc. The factor S may be regarded as a relative measure of the change in the surface activity compared to the initial surface activity of zirconium in a given acid concentration.

An expression for S has been developed by differentiating the equation $W = Ct^n$

$$\text{then} \quad \frac{dW}{dt} = nCt^{n-1} \qquad (5)$$

Substituting for $\frac{dW}{dt}$ in the above equation

$$nCt^{n-1} = kS \qquad (6)$$

and

$$S = \frac{nCt^{n-1}}{k} \qquad (7)$$

where S is a surface activity factor, C a rate constant for the overall reaction at temperature T, t is the elapsed time, n is the slope of the log-log plot of the weight loss versus time and k is the initial corrosion rate taken at $t = 0$ where $S = 1$.

Values for S have been calculated for $t = 48$ hours and are tabulated in Table 3. Assuming a value for $S = 1$ for the initial rate it is seen that the surface activity ranges between 1.88 and 3.28 times the initial activity. At 200 C the activity attains a value 3.62 times the initial activity before the transition to lower corrosion rates occurs. Between 1 hour and about 16 hours at 250 C there is no significant change in the value of S, and hence surface activity. After 16 hours the transition to lower corrosion rates occurs. Also, the values for S at 1 hour and 16 hours are close to unity. These observations suggest that at 250 C the surface initially became rapidly saturated with corrosion sites.

Two possible mechanisms are suggested to explain the increase in surface activity and corrosion rate with time.

1. Intergranular attack which undermines and dislodges entire grains of metal from the surface of the metal.

2. Cathodic inclusions or corrosion sites which may influence the rate of metal corrosion as discussed by Tomashov.[17] The principal feature of this mechanism is the acceleration of the corrosion rate through the growth and formation of cathodic sites. As the area and/or number of cathodic sites increases, the total corrosion current and hence corrosion rate increases, eventually reaching a maximum when the limiting polarization current is attained. Both these mechanisms are discussed below.

Intergranular Attack

Metallographic studies lend support to the explanation that intergranular attack is responsible for the increasing corrosion rate. Intergranular corrosion was observed in the advanced stages of corrosion of equiaxed alpha-annealed and beta-annealed furnace-cooled structures as shown in Figures 7 and 13 respectively. Intergranular attack severe enough to cause possible loss of grains was exhibited only at the higher temperatures

TABLE 7—Summary of Visual Examination of Vacuum Annealed Zirconium After Exposure to 20 Percent HCl At Different Temperatures

Temp. of Test, Degrees C	Conditions of Test	Visual Appearance of Surface of Specimens
No test conducted	3/0 finish and chemical polished prior to annealing at 1150° C for 3 hrs under high vacuum	White metallic luster
150.....	Tested 5 to 25 days after annealing	Yellowish tinted tarnish, metallic luster
175.....	Tested 19 months 21 days after annealing	Yellowish tarnish, metallic luster
175.....	Chemical polished after 19 months 21 days after annealing	Black hydride film
187.5.....	Tested 19 months 21 days after annealing	Yellowish tarnish, metallic luster
187.5.....	Chemical polished after 19 months and 21 days after annealing	Black hydride film
200.....	Tested 5 to 25 days after annealing	Black hydride film. Some grain faces possess black film, others do not. Intergranular corrosion between the Widmanstatten plates initiated.
225.....	Tested 5 to 25 days after annealing	Black hydride film. Some grains coated with hydride others not. Interlamellar corrosion more severe
250.....	Tested 5 to 25 days after annealing	Similar to specimen corroded at 225 C except that lamellar corrosion has penetrated more deeply as shown in Figure 12

TABLE 8—Summary of the Affect of H_2, O_2, N_2, A_2 on the Corrosion Rate of Zirconium At 200 C in 20 Percent Hydrochloric Acid

Initial Atmosphere	Specimen Number	Corrosion Rate Milligrams per sq. decimeter in 48 hrs.	Mils per Year
Test cells evacuated before test	1	518	57
	2	336	37
Nitrogen	1	430	48
	2	456	50
Oxygen	1	516	57
	2	452	50
Argon	1	488	54
	2	490	54
Hydrogen	1	434	48
	2	526	58

and for long test periods as indicated in Figure 7. In addition to the possible loss of grains, intergranular attack progressively exposes a greater area of metal to the acid as well as providing narrow channels and cracks filled with corrosion product, the former tending to accelerate and the latter to hinder corrosion attack with time.

Evidence that the initial surface chemistry controls the reaction indicates that the layer of corrosion products is not sufficiently thick or dense to exert control over the corrosion reaction. There is, however, the possibility that the reaction may eventually become diffusion controlled in this way, thereby offering a possible explanation for the transition to a lower rate of weight loss after 72, 24 and 16 hours at 200, 225, and 250 C.

The facts do not satisfactorily explain why the increase in corrosion rates and surface active factors with time at 150 and 175 C are about the same as those at 187.5 and 200 C when the former exhibits little or no intergranular attack compared to the latter temperatures. Also, the corrosion rate and surface activity increases in the early stages of corrosion, when insufficient time has elapsed for loss of grains by intergranular attack.

The Role of Hydrogen and Cathodic Sites

The Mechanism of Formation of Zirconium Monohydride. A more satisfactory explanation of the acceleration in corrosion rate is the formation of cathodic sites as indicated earlier. The increasing numbers of cathodic sites are believed to be autogenerated by the mechanism described below.

Hydrogen has been shown to have an important influence on the corrosion of zirconium in hot water and hydrochloric acid.[7,13] Active hydrogen supplied by the over-all reaction.

$$Zr + 2HCl + H_2O \rightarrow ZrOCl_2 + 4H$$

is disposed of by adsorption and diffusion into the zirconium lattice, by formation of zirconium monhydride, and by combination and liberation as a gas.

It was shown earlier that the addition of molecular hydrogen to the gas phase in the test cells had no significant effect on the corrosion rate, and presumably on surface processes. This suggests that the effect of hydrogen is controlled by the reaction between hydrochloric acid and zirconium at each temperature investigated. This is reasonable, since although there is an adequate supply of hydrogen ions in the acid solution and molecular hydrogen in the gas phase, neither has a favorable energy relationship for dissociation and adsorption on the surface and the latter is inaccessible to the immersed surface.

It is postulated that the monohydride is formed by the adsorption and diffusion of active hydrogen from the corrosion reaction at micro-cathodic sites, causing them to grow into hydrogen rich, local, discrete volumes or macrocathodes. Cathodic impurity centers would have the effect of increasing the formation and growth of hydrogen cathodic sites, particularly in regions where they might tend to concentrate, such as at grain boundaries. By virtue of their higher hydrogen content, the macrocathodes are more resistant to corrosion attack by the acid. The surrounding anodic areas corrode more rapidly, eventually undermining the less soluble hydrogen charged macrocathodes, which are retained on the surface as particles of a loosely connected porous layer as implied by Figures 6 and 9. Some of the hydride particles are oxidized by oxygen derived from the water, which may explain the presence of white monoclinic ZrO_2 entrapped in the porous layer, the rate of oxidation increasing with temperature as shown in Table 5.

A few milligrams of the white powder were collected from a large number of rinsings of test specimens. Electron microscopy revealed ultimate crystals in a size range well below 0.05 micron as indicated in Figure 18. The assumption that the oxide particles were formed originally from hydride particles would place the ultimate size of the hydride macrocathodes in the same size range as the oxide particles.

When hydrogen is adsorbed into the lattice of zirconium the lattice is expanded, rendering it increasingly susceptible to further hydrogenation as a consequence of higher diffusion rates in metal already charged with hydrogen.[18]

As the area of cathodic sites increases with time, cathodic polarization decreases and the total corrosion current and rate grows, thereby giving rise to an accelerating corrosion rate. Simultaneously, the dissolving anodic areas decrease, the anodic polarization remaining small, but increasing relatively slowly with time. Eventually, however, the anodic current density attains values where intensive polarization develops as a consequence of corrosion products, (e.g., hydrated zirconium oxychloride) forming at a rate faster than can be removed by diffusion and convection and thereby impeding the diffusion of chloride ions through the corrosion products to the metal surface. When this occurs the corrosion current, and rate may be expected to drop off rapidly.

Another effect of hydrogen is to vary the activity of the anodic areas. The overall reaction is the sum total of the rates at anodic and cathodic activation sites; hence, the experimental energy of activation is characteristic of a statistical population of active sites each differing in its activation energy. As the hydrogen concentration at anodic surfaces increases, a change in the activation energy may be expected to occur when the active sites change to a configuration having a different energy of formation for the activated complexes necessary for the release of zirconium ions to the solution. It was found in this study that hydrided zirconium gave a lower activation energy for dissolution in hydrochloric acid.

Thus, diffusion and hydrogen adsorption would be expected to give a lower activation energy if either or both of these participate in the transition to lower corrosion rates. This is supported by the lower activation energies represented by the lower slopes of the upper portions of the Arrhenius curves in Figure 2.

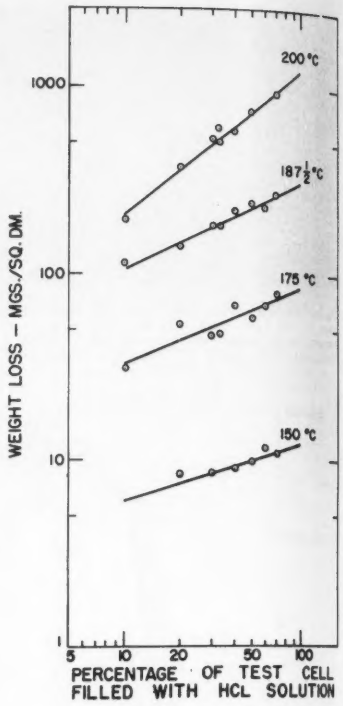


Figure 17—Log-log plot of the percentage of the test cell filled with 20 percent hydrochloric acid solution versus the weight loss in 48 hours at different temperatures.

Another contributing factor for the transition to a lower corrosition rate is the slow increase in the general hydrogen level of the metal, thereby possibly reducing the open circuit corrosion potential and hence corrosion current between anodic and cathodic sites.

The individual or compound effect of the mechanisms described above may explain the sudden transitions in the weight loss versus time curves at 200, 225 and 250 C in 20 percent hydrochloric acid. It follows from the above explanations that the transition should occur earlier at higher temperatures, as was observed. The depletion in acid concentration was calculated from specimen weight losses after 48 hours exposure to 20 percent hydrochloric acid at 150, 175, 187.5, 200, 225, and 250 C giving the values for the final acid concentration of 19.99, 19.98, 19.93, 19.69, 19.39 and 18.83 weight percent hydrochloric acid respectively. The gradual depletion and small change in acid concentration represented by these values does not explain the abrupt transitions observed in Figures 2 and 4. Also, the transition has been observed in iodide zirconium, where the acid depletion is much less than for commercial grade zirconium under similar conditions of test.[19]

Kinetic Considerations. To develop further an understanding of the influence of hydrogen on the temperature

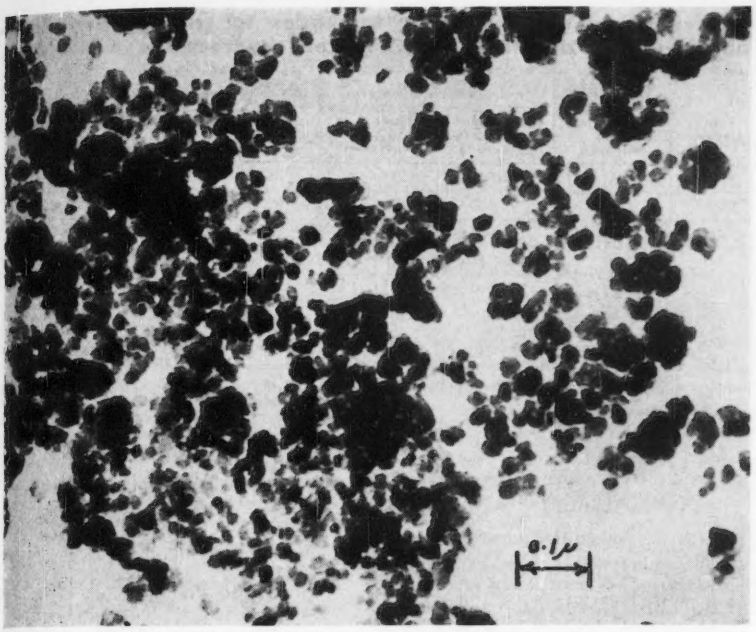


Figure 18—Electron micrograph of aggregates and individual particles of white monoclinic zirconium dioxide exuded by porous black hydride film. Magnification approximately 105,000X. Ultimate particle size appears to be well under .05 micron.

dependence of the reaction, the kinetic theory of absolute reaction rates has been applied.[20] However, the values obtained can only be interpreted in a comparative sense, since no correction has been made for the deviation in acid concentration from the nominal concentration (20 percent) resulting from the use of partially filled test cells.

For a first order reaction it has been shown that the experimental frequency factor A in the Arrhenius equation is related to the entropy of activation ΔS^* by the relationship:

$$A \equiv e.\frac{kT}{h}.e^{\frac{\Delta S^*}{R}} \qquad (8)$$

Where k is Boltzmann's constant, h is Planck's constant, and ΔS^* is the entropy of activation.

The experimental free energy ΔF^*, entropy ΔS^*, and heat of activation ΔH^* may be calculated from the following expressions:

$$\Delta H^* = E_{exp.} - RT \qquad (9)$$

$$\Delta F^* = \Delta H^* - T\Delta S^* \qquad (10)$$

Using the above relationships, values for ΔF^*, ΔH^*, and ΔS^*, uncorrected for temperature dependent phase rule changes in the acid concentration in the test cells, were calculated from the experimental data and tabulated in Table 9 for temperatures of 150, 175, and 200 C.

The large positive entropy of the as-received alpha-annealed zirconium of 74.9 calories/mole/°K is indicative of a relatively higher probability of formation of the activated complex, compared to the beta-annealed zirconium and hydrided zirconium having entropies of 71.4 and 23.4 calories/mole/°K. The smaller entropy for the beta-annealed zirconium is consistent with the reduction in the concentration of active sites brought about by vacuum annealing, through removal of hydrogen, redistribution of impurities, structural changes, and other phenomena. Likewise, the small entropy for the hydrided zirconium is consistent with the smaller probability of formation of the activated complex expected for the surface lattice configuration of zirconium hydride.

Table 9 shows that the entropy of activation or probability factor is relatively unaffected by the temperature, whereas the rate determining factor, the free energy of activation, changes sharply with temperatures.

The increase in the values of the frequency factor and the entropy of alpha-annealed zirconium after 48 hours over the initial values at time zero is interpreted as a measure of the increase in the number of active sites generated by the corrosion surface processes.

Summary and Conclusions

1. The dependence of the corrosion rate of a specific grade of commercial zirconium on temperature, for a closed system ⅓ filled with acid solution and for an exposure time of 48 hours has been represented by an Arrhenius type equation:

$$\log_{10} W = \frac{-E_{exp.}}{2.303\,RT} + \log A$$

A value of 32,359 ± 1606 calories per mole was determined for $E_{exp.}$ for temperatures of 150 to 200 C inclusive and acid concentrations from 5 to 37 percent inclusive.

Elimination of experimental systematic errors resulted in a corrected experimental activation energy of 36,436 calories per mole for a test cell 100 percent filled with acid solution.

2. The dependence of the corrosion rate on time was found to obey the empirical equation:

$$W = Ct^n$$

The values for the exponent n were found to range between 0.98 and 1.43 and the rate constant C between 0.080 and 108 mgs/sq dm/t^n for temperatures between 150 and 250 C inclusive. The increasing corrosion rate was attributed to the growth of cathodic sites autogenerated by the corrosion reaction, which caused an increase in the total corrosion current.

3. The initial rate constants were found to have a temperature coefficient of activity of 31,540 ± 2,860 calories per mole. This value was in substantial agreement with the overall temperature coefficient of activation of 32,359 ± 1606 calories/mole, from which it was concluded that the corrosion mechanism was controlled by the initial reaction up to the transition point.

4. The corrosion of zirconium in hydrochloric acid undergoes a transition from an accelerating to a decelerating rate after a period of time dependent on the temperature. The time for the transition to occur becomes shorter the higher the temperature of the acid solution, being less than 48 hours at 225 and 250 C.

It is suggested that the transition may be an individual or compound effect of intensive anodic polarization and a more gradual effect produced by the increase in hydrogen content of the metal with time. The latter may reduce both the open circuit potential between cathodic and anodic sites and the chemical activity of anodic surfaces.

5. Both delta zirconium monohydride and monoclinic zirconium oxide were identified in the black surface films found on zirconium at all levels of temperature and exposure time employed. It is postulated that the porous hydride layer is formed from hydrogenated macrocathodes undermined by corrosion of surrounding anodic material. The presence of submicron monoclinic zirconium oxide is explained by oxidation of the hydride particles by water.

6. Progressive intergranular corrosion of equiaxed grain structures occurred between 150 and 250 C in zirconium of the particular analysis under study. Recent work has shown that higher purity commercial grade metal does not suffer intergranular attack.[19]

7. Vacuum annealing at 1150 C for 3 hours reduced the corrosion rate without changing the corrosion mechanism, the temperature coefficient of activation being 32,800 calories per mole. Both intergranular and interlamellar corrosion of the Widmanstatten plates were observed.

8. Fully hydrided zirconium metal was found to be superior in corrosion resistance to hydrogen-free metal at tempera-

TABLE 9—Summary and Comparison of Quasi-Thermodynamic Quantities for as Received Alpha-Annealed, Beta-Annealed and Hydrided Zirconium in 20 Percent Hydrochloric Acid*

Condition of Zirconium Metal	Temp. Degrees C	Percent of Test Cell Filled with Acid Solution	Experimental Temperature Coefficient of Activation cals/mole	Frequency Factor $\log_{10}A$ Where A is In mgs/dm²/ 48 Hours	Heat of Activation ΔH^* cals/mole	Free Energy of Activation ΔF^* cals/mole	Entropy of Activation ΔS^* cals/mole/°K
As-received alpha-annealed	150	33	33,635	18.273	32,847	2,697	71.2
As-received alpha-annealed	175	33	33,635	18.273	32,749	149	72.8
As-received alpha-annealed	200	33	33,635	18.273	32,689	—2,711	74.9
As-received alpha-annealed	200	100	36,436	19.9	34,954	—3,046	80.4
As-received alpha-annealed** (initial)	200	33	31,540	15.26 (t = o)	30,594	—2,006	68.9
Vacuum beta-annealed and furnace cooled	200	33	32,814	17.48	31,968	—1,832	71.4
Zirconium hydride	200	33	10,150	6.962	9,165	—2,435	23.4

* Values for quasi-thermodynamic functions are uncorrected for the deviation from the nominal HCL concentration of the liquid phase resulting from partially filled test cells. Data based on 48 hours test period.
** Frequency factor $\log_{10}A$ is 15.26 where A is in mgs/dm²/hour and t = 0.

tures greater than about 180 C, the temperature at which the Arrhenius plots of the hydrided and hydrogen-free zirconium intersect. The temperature coefficient of activation was 10,150 calories per mole and the frequency factor $\text{Log}_{10} A = 6.962$ in 20 percent hydrochloric acid.

9. The addition of molecular hydrogen, oxygen, nitrogen, and argon to the vapor phase resulted in no detectable effect on the corrosion of zirconium at 200 C in 20 percent hydrochloric acid. Active hydrogen, however, released at the surface of the corroding metal, has been shown to have a major influence on corrosion.

10. Unusually corrosion resistant air-formed films may be produced at room temperature on vacuum annealed zirconium by exposure to air for long times. These films exhibit substantially lower corrosion rates and are free of the black hydride coating normally found when zirconium is corroded at 175 and 187.5 C in 20 percent hydrochloric acid.

Acknowledgment

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Any discussion of this article not published above will appear in June, 1960 issue.

Field Service Results of

Silicon Vs Selenium in Cathodic Protection*

By DON P. WILSON

Abstract

The high conversion efficiency of silicon rectifiers for DC output ratings above 30 volts has resulted in substantial savings in power costs. However, silicon failures in high lightning incidence areas and on power supply lines with poor control of switching transients have been unacceptably high. Peak inverse voltage failures of silicon can be caused by lightning induced transients in either the supply lines or in the CP loop without a direct strike. Silicon and selenium failure rates by areas are compared with a Bureau of Standards lightning incidence rate map. Data covering the variation of CP loop resistance from dry anode bed to moist soil condition confirms the wisdom of secondary overcurrent protection. A magamp circuit which controls DC current within plus or minus 2 percent with loop resistance varying from .6 to 2.5 ohms is discussed. Present and future developments to protect silicon against PIV failures are discussed.
5.2.3

Introduction

THE PREDICTED high conversion efficiencies of silicon rectifiers and resultant lower power costs for a specified protective current have been realized in field experience. Unfortunately, however, unacceptably high failure rates of silicon have nullified this gain. As a result, silicon is being used successfully only in those areas which enjoy low lightning incidence rates. A review of fundamentals of rectification will facilitate an understanding of conversion efficiency and an appreciation of silicon limitations.

The time honored practice among corrosion men of calling a complete DC power supply assembly a "rectifier" causes some confusion. Actually, the complete assembly consists of a transformer with means for voltage selection, meters, circuit breaker, and rectifier. The system takes AC voltage from an external source such as commercial power lines or an engine generator, steps the voltage up or down to approximately the desired level, and rectifies it to DC voltage for delivery into the "CP Loop." The term, "CP Loop," is used to define that circuit from the positive terminal of the DC power supply through cable to the anodes, through earth or fluid to the protected structure and through cable back to the negative terminal of the DC power supply. For a specific voltage selected, the current flowing in the CP loop is controlled by the resistance of that loop according to Ohm's law.

In the process of changing AC voltage and rectifying it to DC, three types of voltage or current are involved. Peak voltage, shown in Figure 1, is used for design purposes only but the rms (root mean square) value is measured by conventional AC ammeters and voltmeters. Note that DC values are average values equal to approximately 90 percent or .9 rms values. In electrical power circles, power efficiency is referred to as efficiency but in cathodic protection, conversion efficiency (referred to hereafter as CE) is the criterion. By substituting .9 rms values for DC values in the CE formula and assuming a perfect no loss circuit with unity power factor, it can be shown that the maximum possible CE for single phase circuits is 81 percent:

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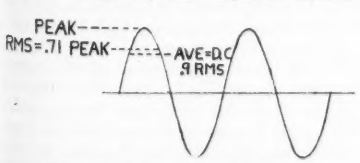


Figure 1—AC waveform (idealized).



About the Author

DON P. WILSON—President of Lester Equipment Mfg. Co., Inc., he became active in the cathodic protection field shortly after joining Lester in 1955. He was among the first to employ silicon rectifers and was identified with their application to well casings in the Shell-Ventura field. A graduate in aeronautical Engineering at Boeing School of Aeronautics, he has been employed in United Airlines, Fairchild Aviation Corp., and Federal Telephone and Radio. As a pilot on active duty in the Navy, he was involved in aircraft radio and electrical installations. He set up the West Coast Division selenium manufacturing operations for Federal Telephone and Radio.

$$\text{Single Phase CE} = \frac{\text{Volts}_{dc} \times \text{Amps}_{dc}\ (\text{output})}{\text{Watts}_{rms}\ (\text{input})} = \frac{\text{Volts}_{dc} \times \text{Amps}_{dc}}{\text{Volts}_{rms} \times \text{Amps}_{rms}}$$

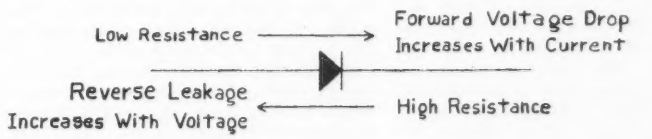
$$= \frac{.9\ \text{Volts}_{rms} \times .9\ \text{Amps}_{rms}}{\text{Volts}_{rms} \times \text{Amps}_{rms}} = \frac{.81\ \text{Watts}_{rms}}{\text{Watts}_{rms}} = .81 \text{ or } 81 \text{ percent}$$

The apparent 19 percent loss results from comparing rms watts input with DC or average watts output, the pulsating nature of which for single phase full wave circuits is illustrated by the lower waveshape of Figure 3. This pulsating characteristic introduces in the DC output a ripple at harmonics of the AC supply frequency,[1] but this ripple is not registered by the DC output meters. It can be shown that the apparent 19 percent loss in a perfect otherwise no-loss single phase full wave circuit is present in the ripple. By insertion of output filters, it is possible to make the DC meters indicate a portion of the power otherwise buried in this ripple, but when one considers the filter insertion loss, the resulting net power is less.

The decision to filter or not to filter becomes a matter of semantics, depending upon what portion of the ripple one recognizes as positive with respect to the protected structure. In any case, losses in every component of the rectifier power supply reduce the actual CE to appreciably less than the theoretical maximum.

Use of Single Diode

In cathodic protection power supplies the rectifiers employed are almost universally solid state semi-conductors which conduct current at low resistance in one direction but present high resistance to the flow of current in the opposite direction. A single diode or cell, as illustrated schematically in Figure 2, is a half wave rectifier. In the case of selenium, this symbol represents one cell or a number of selenium cells connected in series. Note that in the forward or conducting direction there is enough resistance to cause a forward voltage drop which increases with an increase of current conducted. This forward voltage drop decreases with elevation of the operating temperature of the cell or diode. In rectifier circuits these diodes are called upon to conduct and block successively in alternate halves of the AC cycle. Since the resistance of a diode in the reverse direction is not infinite, during the blocking function there is a reverse leakage which increases with increase of the applied



Fwd. Voltage Drop + Rev. Leakage = Losses In Rectifier
= Reduction C.E. Below 81% Or 96%
= Heat Dissipated By Rectifier

Figure 2—Schematic of single diode.

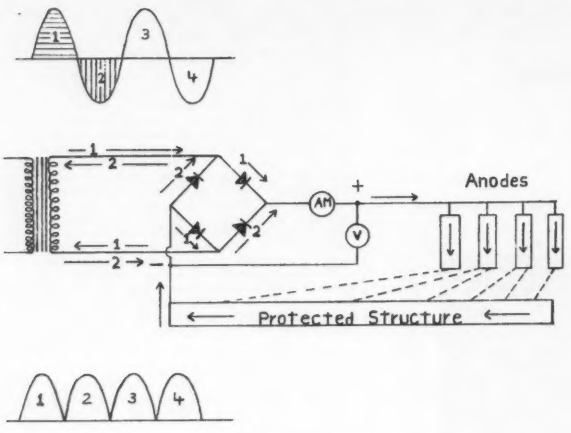


Figure 3—A typical single phase full wave bridge circuit connected to the CP loop.

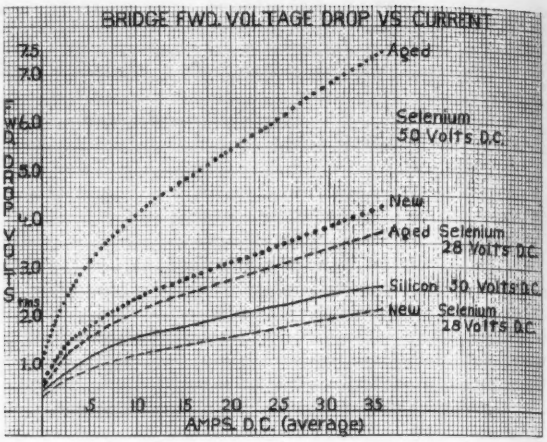


Figure 4—Bridge forward voltage drop vs current.

voltage. In this blocking function, a diode must block the peak voltage which is termed "peak inverse voltage." In the case of silicon, this characteristic referred to hereafter as "PIV" becomes an important consideration.

Heat generated in the diode by forward voltage drop and reverse leakage must be dissipated by the rectifier assembly. Heat generated in the course of transformation and rectification is a loss. Thus the losses from forward voltage drop, reverse leakage, and transformer efficiency constitute the major part of the difference between the theoretical maximum and actual conversion efficiency.

Figure 3 illustrates a typical single phase full wave bridge rectifier circuit connected to a CP loop. In the first half of the AC cycle, the lobe designated as No. 1 at the top of Figure 3, current flows in the direction of No. 1 and unnumbered arrows to produce the first positive pulse, illustrated as No. 1 at bottom left of Figure 3. Note that No. 1 diodes are conducting and No. 2 diodes are blocking during this half of the cycle.

In the second half of the AC cycle, No. 2 lobe at top left of Figure 3, current flows in the opposite direction through the transformer secondary, approaching No. 2 diodes in their conducting direction and following the same unnumbered arrows as in the first half cycle to produce the second positive puse, No. 2 at bottom left of Figure 3. During this second half of the AC cycle, No. 1 diodes are blocking. Thus a full wave rectifier is essentially a flip-flop device which accepts AC current from two different directions alternately but delivers unidirectional or direct current as a series of positive pulses. The pulsating nature of the resulting voltage and direct current produces the ripple mentioned previously. Note that in a single phase bridge circuit the voltage suffers forward voltage drop through two diodes.

Most of the electrical characteristics of solid state semi-conductors are non-linear and vary between otherwise identical diodes. The relatively high conversion efficiency of silicon vs selenium stems primarily from the higher voltage capability of a single silicon diode over that of a single selenium cell. Although selenium is now available with 45 volts rms input rating, there is reason to question its service life until more experience has been gained. Consequently, this comparison is limited to 36 volts rms input selenium cells which are capable of 28 volts DC output from a bridge of one cell per arm, after allowance for expected increase of forward voltage drop over a ten year period.

If higher DC output voltage is desired from selenium, it is necessary to use two or more cells in series in each of the four arms of a single phase bridge. A plurality of cells in series increases the forward voltage drop by a factor equal to the number of cells in series per arm. In contrast, silicon power diodes are available with PIV ratings up to 500 volts which converts to 355 volts rms input. However, because silicon is vulnerable to peak voltage transients, the maximum DC output from a bridge of 500 PIV diodes should not be more than approximately 130 volts DC. Since selenium forward voltage drop increases with age, any comparison of selenium vs silicon must consider both the new and aged conditions. Theoretically, silicon does not show any change of electrical characteristics with age as results over the four years of silicon availability confirm. Selenium aging is nonuniform as evidenced by the fact that some selenium stacks experience about 80 percent of their expected forward voltage drop increase in the first year of continuous operation whereas others seem to age uniformly throughout their 10 to 15 years of useful life. There is no unanimity of opinion among authorities in this field as to the exact explanation for this difference in aging. In any case, the end of useful life of selenium is reached when final increase of forward voltage drop precludes obtaining the desired DC output voltage.

In general, the forward voltage drop of a new selenium cell is less than that of a comparable silicon diode, but in the course of aging it loses this advantage as illustrated in Figure 4. In Figure 4 a silicon bridge with 50 volts DC output capability is plotted against two selenium bridges in the new and aged conditions: one with 28 Vdc output and one with 50 Vdc output. Note that for DC output voltages requiring two or more selenium cells in series per arm, silicon has a pronounced advantage, as illustrated by the 50 Vdc curves. As an additional advantage, the reverse leakage of silicon is less than 10 percent of that of a well formed selenium cell. Consequently, in over-all conversion efficiency silicon begins to show a slight advantage over new selenium at 15 Vdc and this advantage improves with increase of DC output voltage rating. In Figure 5, illustrating this relationship, the sawtoothed portions of the selenium curves indicate the DC output voltages at which an additional selenium cell is required in series in each of the four arms of the bridge. Figure 6 sets forth conversion efficiencies measured on 639 units, both selenium and silicon, in factory production tests.

As is so often the case in the field of science, gains in certain characteristics are achieved at the price of limitations in other aspects. As mentioned above, silicon is much more prone to failure under voltage transients than is selenium. This may be attributed in part to the fact that at 60 cycles selenium has approximately .02 mfd distributed capacitance per square inch of cell area whereas the small contact area silicon junction has practically none. It is possible that this capacitance of selenium partially absorbs and flattens out voltage surges with a resultant sputtering of impurity spots in the outer portions of the cell. A selenium rectifier stack will usually continue operating after such sputtering, but the violation of the protective paint seal permits ingress of corrosive influences which shortens its life. Silicon, however, fails instantaneously when its Zener voltage level is exceeded. This Zener voltage level is usually somewhat higher than a particular diode's PIV rating, depending upon the safety factor specified by the particular diode manufacturer for quality control. This vulnerability of silicon to voltage surges above rating has proven to be its principal limitation to successful application in cathodic protection.

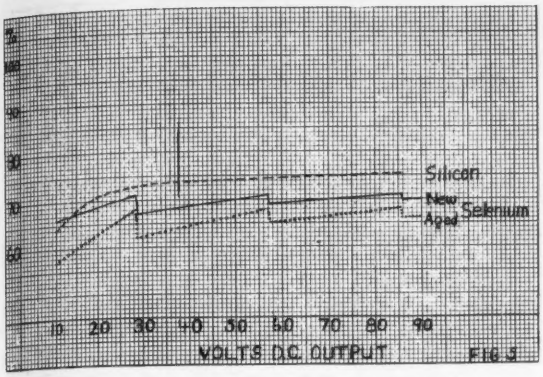


Figure 5—Conversion efficiency (single phase bridge).

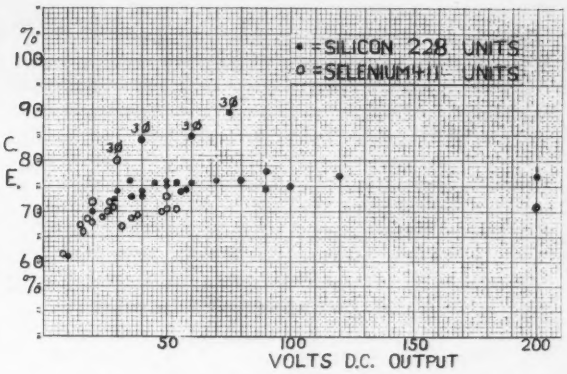


Figure 6—Conversion efficiencies of silicon and selenium.

Figure 7 illustrates a circuit in which capacitance and impedance are introduced externally to the silicon bridge in an effort to provide some small level of protection to high voltage surges. Unfortunately, it has not yet been possible to set up a test project under controlled conditions in any of the high voltage laboratories of the country to establish effectiveness of such an approach. All lightning arresters known to the author are too slow-acting to protect silicon.

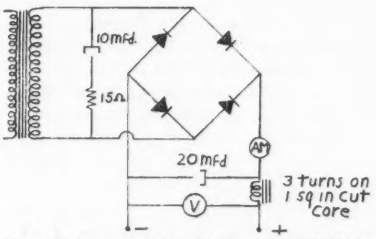


Figure 7—Values for 4-1-1 bridge of 200 PIV diodes 50 volts DC output.

Deterioration of Selenium

Many corrosion engineers do not realize that selenium deteriorates on the shelf without voltage applied. In the manufacture of selenium cells, prior to their assembly into stacks and painting, they are "formed" electrically by pulsating DC to produce high resistance to the flow of current in the reverse direction. This reverse resistance deteriorates when stacks are left without applied voltage, thereby increasing the reverse leakage current to produce more than normal heat when voltage is ultimately applied. Frequently, if full rated AC input voltage is applied after an extended period of idleness, the selenium cells will sputter around the outer areas, thus breaking the protective paint seal. Consequently, after storage or installation shut-down, it is better to bring the AC input voltage to the rectifier up to rating gradually over a 15 minute period. This reforms the cells and decreases the reverse leakage gradually before it reaches sufficient level to burn out or sputter localized hot spots. Even installations operating continuously at substantially below their rated DC output voltage will tend to "form" down to the continuing operating level. To prevent this, approximately every three months the rectifiers should be tapped up gradually to their rated volts DC output after removing either the anode or protected structure lead to prevent excessive current. Conduction of current through an external DC load circuit is not necessary for such a reforming exercise.

In allowable operating temperature silicon has an advantage over selenium at the price of a critical limitation. The limiting temperature varies from 150 to 200 degrees C with silicon diodes of different manufacturers, but when this junction temperature is exceeded, the diode fails instantaneously. For long life selenium temperature should not exceed 65 C but higher temperature only shortens its life if the temperature does not reach the flow point of the front electrode alloy. Thus in a properly designed rectifier power supply with adequate de-rating and convection cooling for the maximum ambient temperature specified, exceeding the maximum continuous current rating is the most common cause of short rectifier life.

In the manufacture of selenium cells and silicon diodes 100 percent quality control is economically impractical but somewhere between 1 percent and 2 percent failures in the first year appears to be a reasonable expectancy. In general those that get through the first year should be good indefinitely in the case of silicon or for 10 to 15 years with selenium, provided the voltage and current ratings are not exceeded.

The vulnerability of silicon to excessive current has given impetus to the development of means for close, fast-acting overcurrent protection which has in turn been applied to selenium for enhanced life expectancy. It is well known that CP loop resistance varies considerably with moisture content of the soil. Thus in geographical areas of long dry spells a factor of 4:1 before and after the first heavy rainfall following a long dry spell is quite common. This is, of course, more pronounced with shallow anode beds than with deeply buried anodes. With a relatively fixed DC voltage output from the rectifier, a sizable drop in CP loop resistance following rainfall brings Ohm's law into action to produce an increased flow of direct current, often exceeding the current limit of the rectifier for the ambient temperature.

Figure 8 illustrates a circuit with a three pole circuit breaker, the third pole of which is a relay trip with current sensing element between the transformer secondary and the rectifier input. This circuit provides equally close primary protection for either of two alternate input voltages and over-current protection to the semi-conductors, regardless of voltage selector settings. This has proven very satisfactory for compressor station,

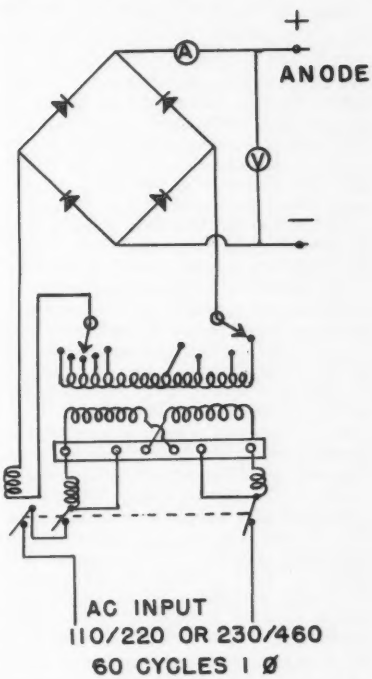


Figure 8—Circuit with a three pole circuit breaker.

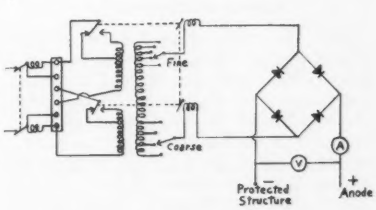


Figure 9

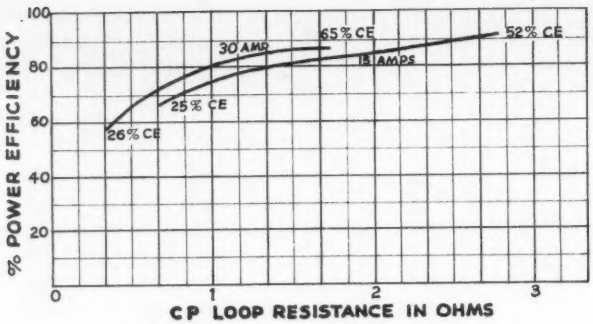


Figure 10—Constant current rectifier.

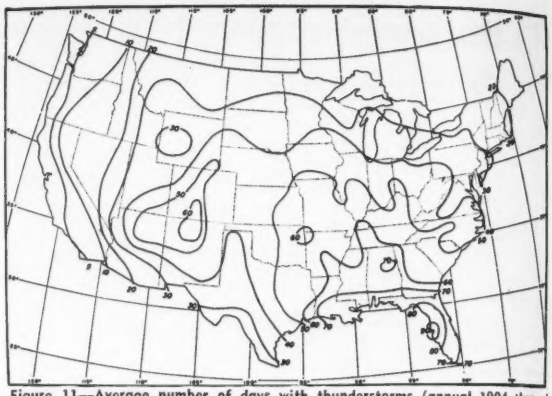


Figure 11—Average number of days with thunderstorms (annual 1904 through 1943). Findings were based on 217 first-order stations.

tank bottom, and oil string casing installations where there are personnel in attendance to detect the shut-down, reduce the voltage selector settings, and turn the unit on again. However, for pipeline installations which are inspected infrequently by lineriders, the possibility of no protective current after an automatic shut-down until the next inspection could result in a pipeline failure. For such cases the Figure 9 circuit offers advantages. When the rated DC current is exceeded within the tolerance of the circuit breaker, the current sensing elements in the secondary actuate auxiliary switches which cut in an overwind on the transformer primary, providing continued operation at lower output voltage and lower current. The rectifier continues to operate in this condition until a linerider reduces the voltage selector settings and resets the circuit breaker to normal position.

For installations subject to regular continuing changes of CP loop resistance the magnetic amplifier regulated constant current rectifier power supply offers more fully automatic operation with the penalty of relatively low CE under certain conditions. Figure 10 illustrates the spread of CE for two current settings of such a unit which can be set for any current between 15 and 30 amps, maintaining that setting within 5 percent with CP loop resistance ranging from .3 to 2.8 ohms within the limitation of maximum 50 volts DC available.

Silicon vs Lightning

With the effort expended to protect silicon against excessive currents, the early silicon cathodic protection installations operated well at high conversion efficiencies until in the summer of 1957. At that time lightning struck a compressor station at Topock on the California-Arizona border, igniting a waste gas pipe. Several days later it was determined that the silicon rectifier had failed but it was impossible to establish when the failure had occurred. Shortly afterwards, a silicon rectifier failed in the San Joaquin Valley without any known lightning activity. Finally on October 20, 1957 southern California experienced a very rare intense lightning storm and within three days 12 silicon failures were reported. It was impossible to inspect all silicon installations immediately after the storm, but it was reasonable to attribute these 12 failures to lightning. In a effort to test the validity of the lightning cause assumption over a wider geographical base, a field survey was conducted.

It was extremely difficult to obtain complete data on both selenium and silicon installation failure history and consequently the selenium data are limited to the 411 units which the writer is in a position to monitor periodically. Within the first year after installation two of the selenium units had failed and a third selenium stack showed signs of early deterioration but this was corrected by cleaning the stack buss connections. At about the same time 20 additional units began to blow circuit breakers sporadically. Removal and testing of the selenium stacks revealed that each one had one or two cells which had developed high reverse leakage at their full input voltage rating. All of the foregoing selenium difficulties were experienced in one oilfield installation at which the AC supply line voltage varies plus or minus 15 volts regularly. No other selenium difficulties were experienced in any of the other installations.

The silicon part of the survey covered a total of 316 silicon installations maintained by five large operators in 18 states. A total of 171 failures were tabulated at 76 installation sites, 23 of which suffered failures from 8 to 2 times. This data becomes more significant when broken down geographically. Two hundred and seven of the installations surveyed were located in California where a total of 14 failures were recorded, 13 of them after known lightning activity. In Figure 11 it will be noted that California has the lowest lightning incidence rate of the country along with Oregon and Washington. The balance 109 silicon installations in 17 states through the southwest, south central, southeast, and eastern seaboard states experienced 157 failures. Silicon was replaced in 23 of these installations more than once, the highest being eight times at one installation. The highest failure rates occurred in East Texas, Louisiana, Alabama, Mississippi, Georgia, and Kentucky. It is not just a coincidence that these same states have the highest lightning incidence rate, as set forth in Figure 11.

National Bureau of Standards Handbook 46, "Code For Protection Against Lightning," contains a very good summary of research on the origin, characteristics and effects of lightning. Of particular importance to this investigation is the fact that voltage surges of 1000 to 1500 kilovolts can be induced on overhead transmission lines by either electrostatic or electromagnetic induction without a direct strike. Although no research on voltage surges induced similarly in pipelines, tanks, and compressor stations are known to the author, it is reasonable to assume that voltage surges of a high order could be measured. One field man reported taking pipe to soil measurements during distant lightning activity and noting end scale fluctuation of the needle at the time of the flashes.

Many of the silicon manufacturers and equipment manufacturers are doing considerable investigative work in search of a cure. It is hampered by the fact that a failed silicon diode does not exhibit any differentiating characteristics attributable to the cause of failure. Most diodes fail as a short although a few have been found open circuited. It is a simple matter to reproduce over-voltage failure of a silicon diode in the laboratory because when the applied voltage exceeds the Zener level for that diode, the reverse leakage current avalanches to burn out the silicon junction. There is no fixed pattern to corrective action to date. In some cases, increase of diode PIV ratings has been followed by long operation but in just as many others the high PIV ratings have failed just as rapidly.

It is doubted seriously that the highest silicon diode PIV rating available today can withstand the effects of lightning induced voltage surges. One operator claims good results over a short time base for a circuit similar to Figure 7, but diodes can be ruined in the laboratory by applying voltage surges to such a circuit at either the AC input or DC output side.

Until further progress can be made in means for protection of silicon, it seems advisable to use selenium in all high lightning incidence areas.

Reference

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Bacterial Activity at the Bottom Of Back-Filled Pipe Line Ditches*

By JOHN O. HARRIS*

Abstract

Bacterial activity at the bottom of back-filled pipe line ditches was compared with that found in normal undisturbed soil. Topics discussed include the effect on bacterial development of: availability of food materials in soil, water availability in soils, effect of oxygen content of soil, soil acidity, temperature variations, application of cathodic protection, compaction and settling of back-fill. A study was made of the role bacteria play in the deterioration of pipe line coatings. The significance of high moisture in soil adjacent to the lines, the moisture content of coatings and the presence of aerobic bacteria near the pipe lines was discussed.

3.3.4

Introduction

A STUDY was made recently of the role of soil bacteria in the deterioration of pipeline protective coating materials. It was apparent at the outset that the pipeline ditch was composed of several different soil zones as opposed to the normal undisturbed soil of the right-of-way. Field observations recorded from many sources led to the concept of four different environmental situations.

Shown in Figure 1 these represent (A) the compacted area on the surface of the back-filled ditch, (B) the semi-dense area above the pipe, (C) a high moisture area immediately surrounding the pipe at the bottom of the ditch, and (D) undisturbed soil in the right-of-way.

It would be expected that these differing environmental situations would have a major influence on the bacteria of the soil. The results to be reported in this discussion regarding the soil moisture content and the numbers of bacteria present at different levels will confirm the concept that the back-filled ditch represents a "man-made" disturbance of the natural situation in the soil. Furthermore, the data to be presented show the environmental conditions in the pipe line ditch are favorable for microbial activity and that this is reflected in the distribution of the bacterial population in the soil of the pipe line ditch. The numbered locations (1-2-3-4-V-VI-VII) in Figure 1 illustrate where soil samples were taken at each bell hole opening. Samples 1 to 4 represent results from the back-filled ditch and V to VII soil from the undisturbed part of the right-of-way.

Bacterial Numbers In "Normal" Soil

In order to form the basis of comparisons, the typical normal soil situation should be understood as an ever-changing equilibrium between the activities of bacteria and other forms of life in the soil and the factors of the environment.

The total number of bacteria in a soil usually reflects the availability of the food supply. Thus in undisturbed pasture or forest soil such as found along a pipe line right-of-way, the majority of the microbial population is found on the surface of the soil in contact with the decomposing organic matter and plant residues. In cultivated soils, the cultivated layer contains the bulk of the population. Under these normal conditions relatively fewer microorganisms are found at depths of one foot or more under the surface of undisturbed soil. At depths comparable to the bottom of the pipe line ditch the total numbers of microorganisms are usually much less than one percent of the numbers near the surface.

These concepts have been well established in the field of soil microbiology during the past 75 years.[1] Likewise the influence of various environmental factors has been studied under many soil conditions. The most important of these soil factors will be considered below.

Available Food Materials

The food for soil microorganisms normally comes from decomposing plant and animal residues in the soil. Many studies[1] have shown that the addition of sugars, straw, fertilizers or other easily decomposed organic matter causes an increase in the number of bacteria living in that soil. The findings of Bushnell and Haas[2] reveal that the presence of hydrocarbons in a soil will support a heavy growth of bacteria capable of utilizing these hydrocarbons as food.

Bacteria usually make use of only a small fraction of the energy available to them in organic matter. The easily decomposed organic matter disappears from a soil most rapidly and as the food supply diminishes the population numbers also decrease. The residual organic matter or humus in the soil represents a fraction relatively resistant to microbial breakdown.

The relationship of food supply to population size at considerable depths in the soil is well illustrated by a phenomenon known as the rhizosphere effect.[3] There are many more bacteria living in the soil adjacent to and in contact with plant roots than are present in the soil a few inches away from the roots. The evidence is conclusive that the root furnishes organic

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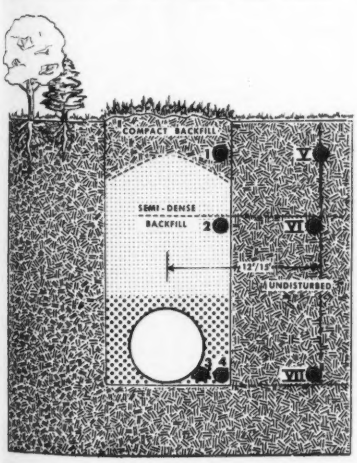


Figure 1—Soil zones in pipeline ditch. This diagram is a composite of photographs and observations of bell-hole openings.

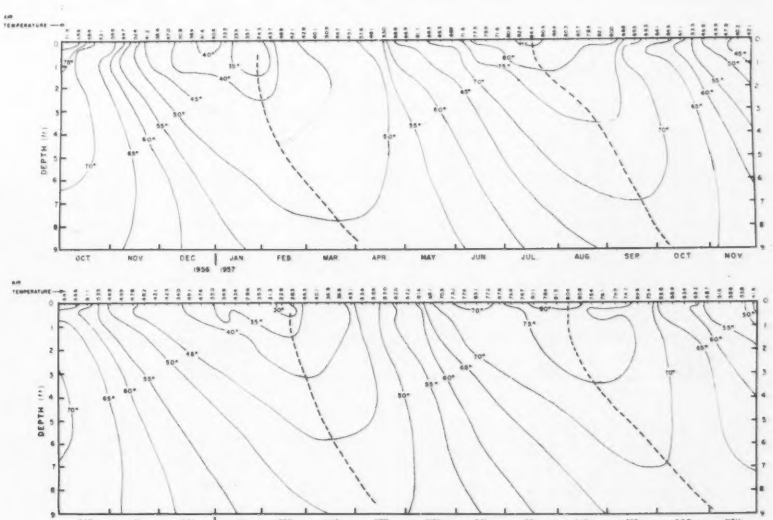


Figure 2—Average weekly soil temperature at Manhattan, Kansas. Data courtesy of Dr. L. D. Bark, climatologist, Kansas Agricultural Experiment Station.

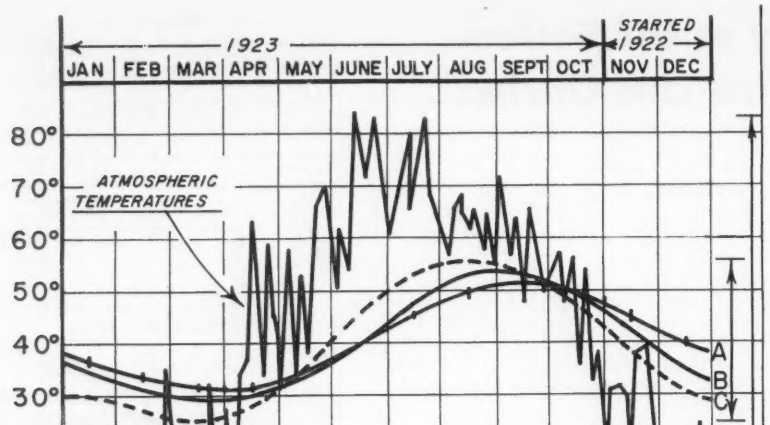


Figure 3—University of Saskatchewan soil survey.

matter as food for bacteria, allowing a higher population to exist than in a normal soil.

Water Relationships

Soil bacteria need water to grow and develop. The actual water needs of most bacteria have never been definitely established but would differ with different types of organisms. The actual availability of water in a soil is more important than the total amount in a soil. Clay type soils hold water much more tenaciously than sands and hold much more water before becoming saturated. The water held by capillary forces is the most important of water to soil bacteria although movement of free water through a soil by percolation is also important in maintaining a moist environment for bacteria. Thus the nature or composition of a soil is directly related to the water relationships with soil microorganisms.

It can generally be assumed that where enough water is present in a soil to support plant growth, adequate water also is present for bacteria. The lower limits for bacterial activity are below the water requirements for higher plants. There are a number of microbial processes that proceed rapidly in dry soils.[1] The upper limit of water concentrations in soil affect bacteria only by the exclusion of soil gases as water occupies the available space in a soil. Thus the most significant result of high moisture content is the lack of oxygen which favors the development of anaerobic bacterial types.

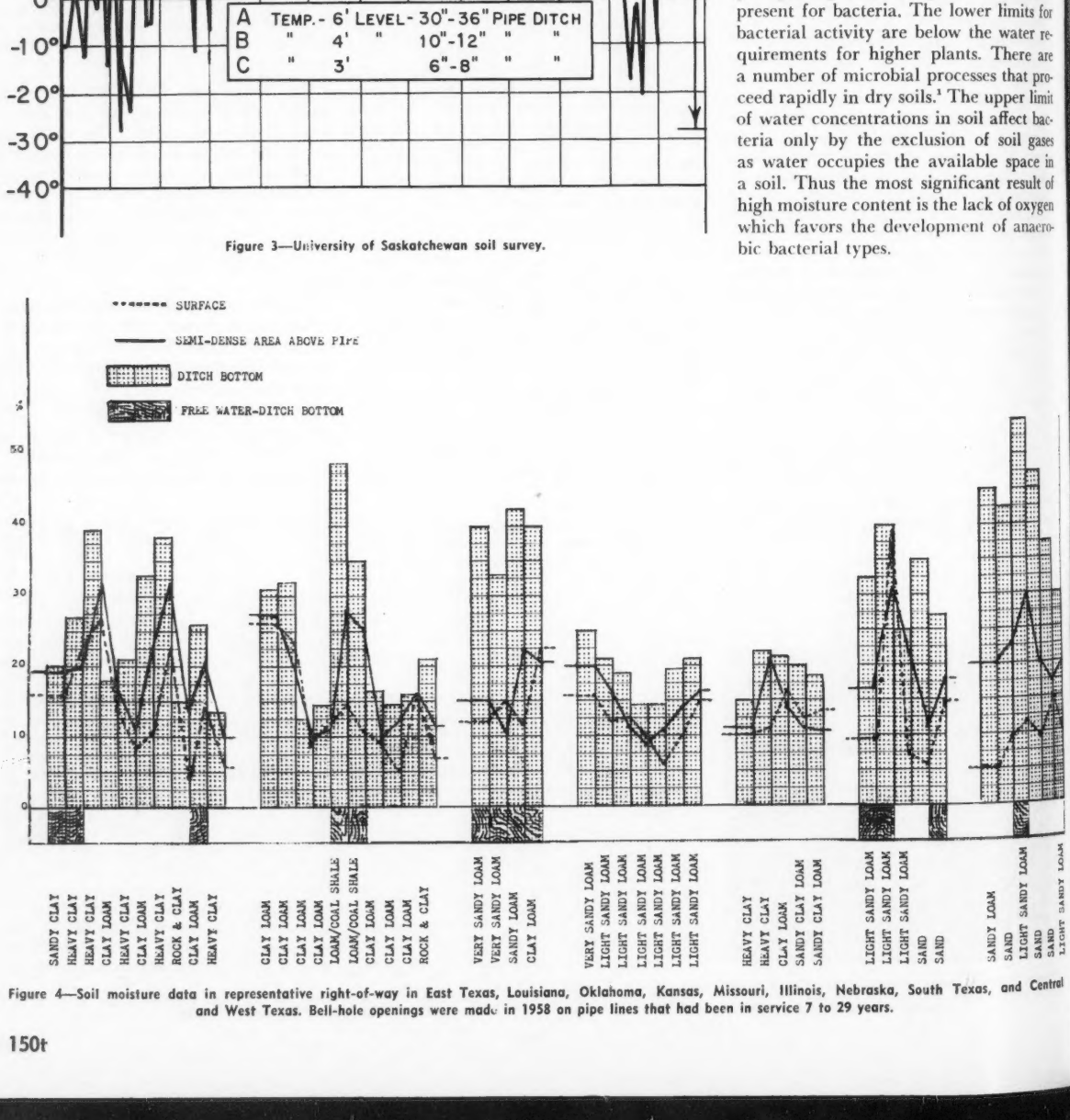


Figure 4—Soil moisture data in representative right-of-way in East Texas, Louisiana, Oklahoma, Kansas, Missouri, Illinois, Nebraska, South Texas, and Central and West Texas. Bell-hole openings were made in 1958 on pipe lines that had been in service 7 to 29 years.

Oxygen and Bacterial Development

Oxygen is important in the energy relations of all cells. Except for photosynthetic forms, all bacteria get their energy by chemical oxidations. The food oxidized may be organic or inorganic. Organic compounds furnish the food for the majority of bacteria in the soil although the inorganic salts used by soil bacteria as energy are important in soil processes. As food materials are oxidized a simultaneous reduction reaction occurs. With aerobic bacteria which require O_2, organic matter is oxidized and oxygen is reduced to water in the respiration process.

Anaerobic bacteria grow in the absence of oxygen. In fact, oxygen is toxic to many anaerobes. Anaerobic bacteria also oxidize organic matter and reduce a variety of both inorganic and organic substances. The typical sulfate reducers[1] represent anaerobes for which much has been published as significant in acceleration of corrosion of bare steel surfaces in the soil. Various carbohydrates, organic acids, proteins, fats, alcohols, and other organic compounds may be reduced under anaerobic conditions.

A third type of microorganism may be facultative with respect to oxygen. Where conditions are aerobic it uses oxygen but when oxygen is limited it grows as an anaerobe. These facultative forms are present in large numbers in undisturbed soil in the right-of-way and also are found in greater numbers in the soil surrounding the pipe at the bottom of the pipe line ditch.

Soil Acidity

Most soils fall in the range of soil acidity where the majority of bacteria are active. The reaction (pH) of the soil has a marked effect on certain crop development and somewhat less effect on microorganisms. Only under extreme conditions of very high acidity or alkalinity in soil would great population changes occur. The pH of soil samples at ditch bottom in twelve states shows little variation.

Temperature Effects

The soil has a marked temperature buffering effect with the greatest temperature changes occurring on the soil surface. The temperature variations of the soil are much smaller than the seasonal variations of the air above it. Furthermore soil temperature changes show a definite lag behind the air temperature.

Figure 2 shows the average soil temperature at depths from 0 to 8 feet and the 2-year air temperature recorded at the Manhattan, Kansas Agricultural Experiment Station. Although the air temperatures showed a variation of average 14.2 to 83.6 F, the soil at 4 foot depths remained between 40 and 72 F.

Similar data are shown in Figure 3 giving the results of a University of Saskatchewan soil survey made in 1922-23. The microbial population of the soil is very diverse in its temperature relations. Some types prefer hot, some prefer cold conditions. The majority have a wide range of growth temperature and would remain active over the range of temperatures recorded in Figures 2 and 3.

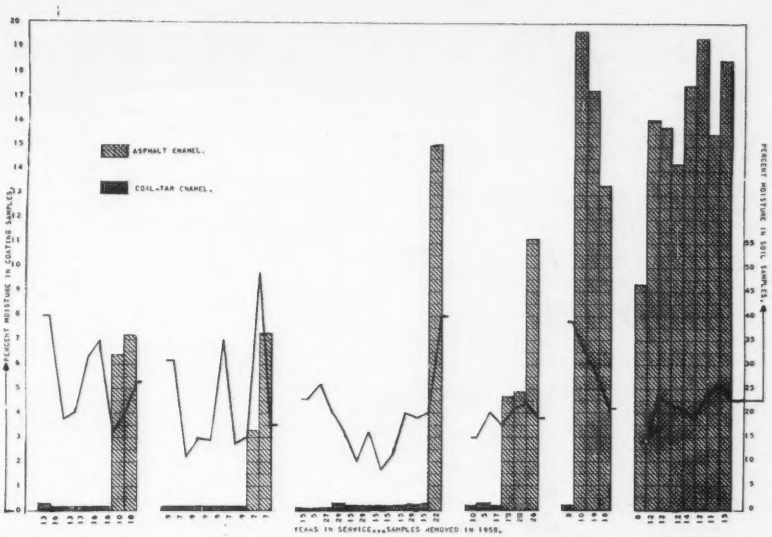


Figure 5—Percent moisture in coating samples removed from pipe and soil samples immediately surrounding the pipe. Data were taken from representative samples of average rights-of-way in North Central and South Central United States. Service time varied from 5 to 29 years.

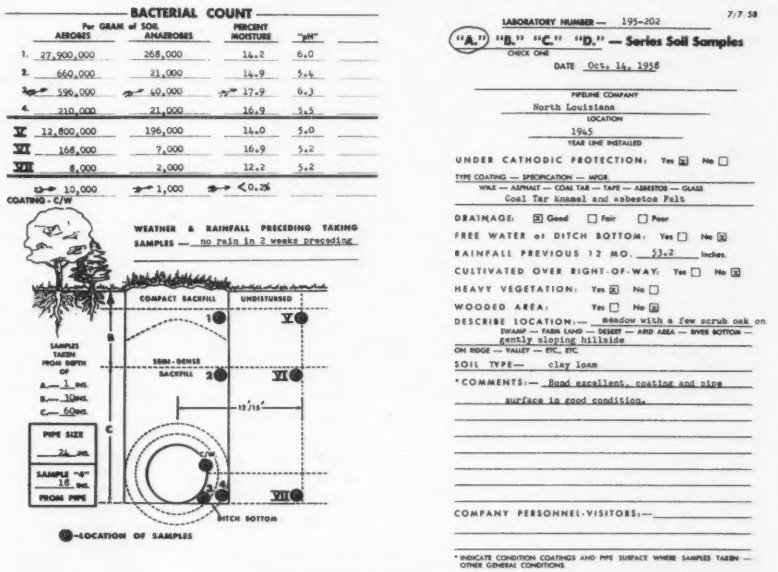


Figure 6—Typical data for bell hole opening.

These factors of the soil environment which influence bacterial growth have been discussed briefly because alterations in the environment may give clues to the microbial activity in the pipe line ditch. The undisturbed soil of a pipeline right-of-way represents essentially equilibrium conditions. It is often cleared of trees in wooded areas, and may be grazed or irrigated or used for cultivated crops depending upon the adjacent land usage. Thus the microbiological situation would be equivalent to the normal situation resulting from the interplay of factors just discussed. The pipe line ditch on the other hand represents a definite alteration of normal soil conditions.

The Back-Filled Ditch

Listed below are some of alterations of the normal soil environments occurring during pipe line construction.

1. Changes in structural units of the soil resulting from the ditching, back-fill operations, and effects of exposing subsurface soil to drying.

2. Compaction and settling of the backfill. This varies with time and with the type of soil. Although the surface may become compacted, most of the zone above the pipe never becomes dense and compact as the undisturbed soil. This influence of the mixing, aeration, settling,

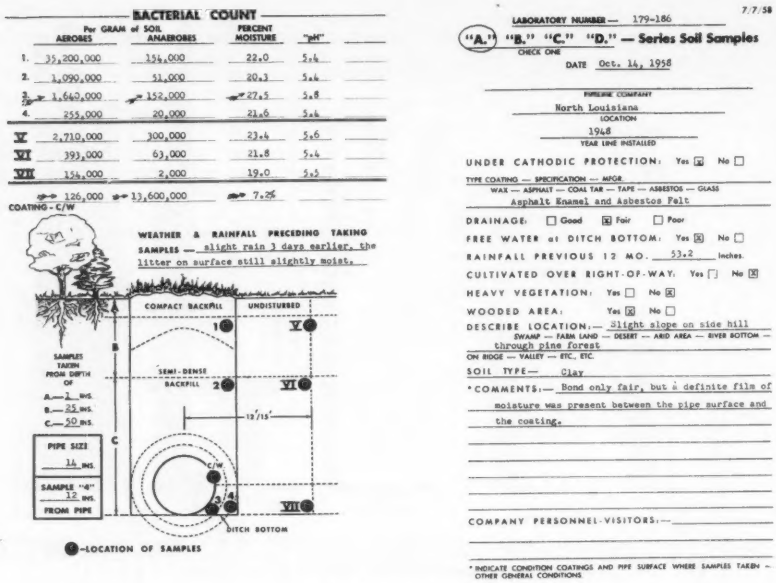


Figure 7—Data for soil samples.

etc. can be observed in some cases for hundreds of years.

3. Mixing the surface and sub-surface soils during backfilling. This will distribute the surface microorganisms throughout as well as surface layers of organic matter of the soil.

4. Large amounts of organic matter added to the environment of the ditch. This organic matter is the coating and wrapping material applied to the exterior of the pipe. This constitutes potential food for soil microorganisms.

5. Cathodic protection applied to the pipe. This may be an additional complicating factor in the altered environment. Certainly the electrical potential would have an effect on the ions of the soil solution and upon soil moisture. This effect would be magnified if the coating resistance were lowered. Most engineers believe that electroendosmosis of water occurs as the result of cathodic protection. This represents a flow of water toward the lines and may account for more moist conditions near the pipe surface and in the ditch bottom.

6. Relationship of cathodic protection to the soil bacteria. Little is known about this matter although moisture changes and pH alterations resulting from cathodic protection could affect organisms in the soil.

7. Soil moisture (water) relationships in the back-filled pipe line ditch as differing from the undisturbed soil of the right-of-way. Because of differences in compaction (soil pore space) it would be expected that aeration would be more complete in the ditch than in normal soil.

Research At Kansas State University

A project has been in progress at the Kansas Agricultural Experiment Station designed to evaluate the actual role that bacteria may play in deterioration of pipe line coating. Four fundamental questions form the basis of this research:

1. Are bacteria able to grow on the coating material currently used on pipe lines?
2. What are the environment conditions of the pipe line ditch and will these conditions allow microbial development?
3. Do bacteria damage coatings by methods which are detectable by physical, chemical, or electrical methods?
4. What is the relation of water content of coatings, soil moisture, microbial activity and cathodic protection?

Data have been published regarding the first question; abundant growth occurs on certain coating materials in the laboratory.[4] Using a large number of pure cultures of bacteria from pipe line soils at ditch bottoms and other sources, tests were carried out on commercially used coating materials. Asphaltic and wax coatings, polyethylene and polyvinyl plastic tapes would serve as food materials for many pure cultures when these were present in an otherwise inorganic culture. This abundant growth was not observed on coal tar coatings. Research is continuing on this phase of the project.

More than 850 soil samples from pipe lines have been studied to learn a few

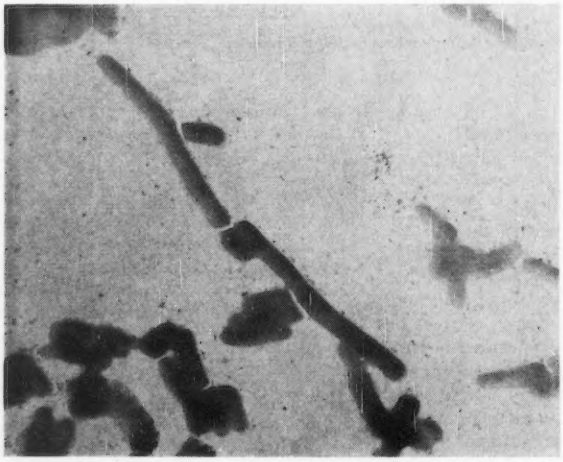
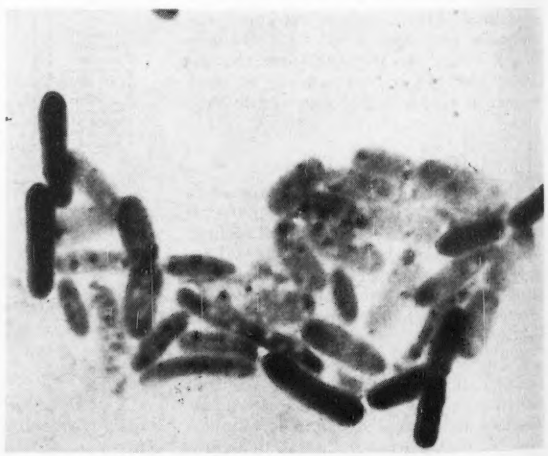


Figure 8—Rod shaped bacteria which tend to form chains in back-filled pipe line ditch. Approximately 7,800X.



Bacterial Count and Moisture Content

Soil Sample Next to Pipe at Ditch Bottom			Coating Sample		
Aerobic	Anaerobic	% Moisture	Aerobic	Anaerobic	%Moisture
342,000	28,000	6.5%	3,000	3,000	<0.2%

Sandy caliche—Drainage fair—Arid—Drainage area from oil field—High salt H_3O.

Figure 9—*Pseudomonas* species isolated from soil in southwest Texas. These bacteria were found adjacent to pipe line coated with coal tar enamel, glass and asbestos felt. Approximately 9,000X.

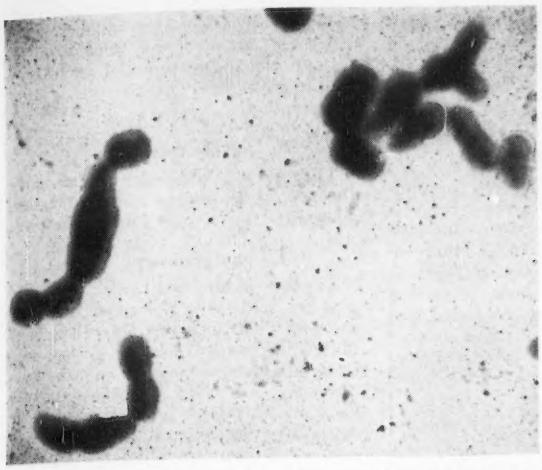


Figure 10—Irregular rod-shaped cells classified as soil forms of *Mycobacterium* or *Corynebacterium* species. In these bacteria, branch forms may be seen together with swollen and club shaped rods. Approximately 17,500X.

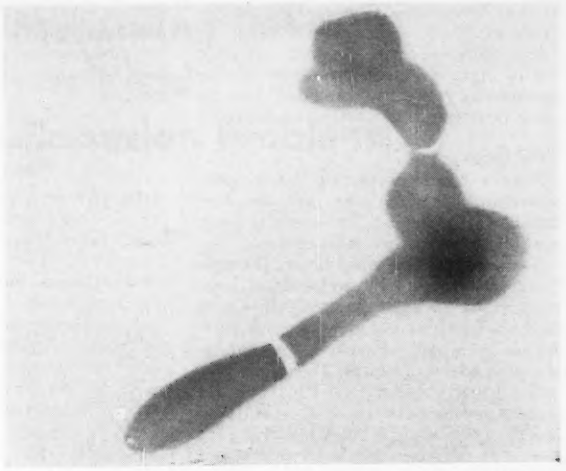


Figure 11—Irregular rod-shaped cells isolated from soil adjacent to coated pipe. Approximately 42,000.

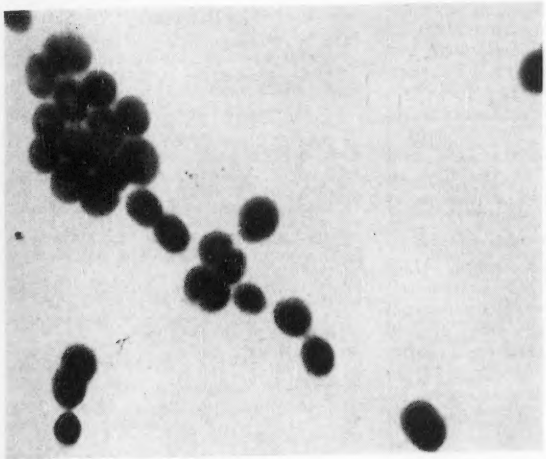
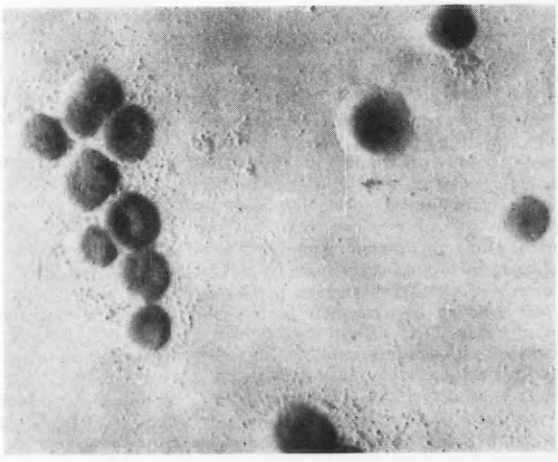


Figure 12—Coccus type bacteria isolated from soil adjacent to pipe line. Approximately 6200X.



Bacterial Count and Moisture Content

Soil Sample Next to Pipe at Ditch Bottom			Coating Sample		
Aerobic	Anaerobic	% Moisture	Aerobic	Anaerobic	% Moisture
66,700,000	2,230,000	32.5%	2,400,000	870,000	19.7%

Sandy loam—Cathodic protection—Free water in ditch—Platte River bottom land—Native grass—Poor bond—Many holidays—Separation felt and asphalt.

Note: This same type of Coccus was found in practically all of the 850 samples secured in 1958-1959.

Figure 13—Coccus type bacteria *(Micrococcus paraffinae)* isolated from soil adjacent to asphalt coated and wrapped pipe line. This electron miscroscope picture of bacteria shadowed with palladium shows the surface and central structure of cell. Approximately 9700X.

facts about the pipe line ditch as an environment. Some of this information has been published.[5] Extension of these data and additional results of these analyses form the main topic of this discussion. When sampling bell hole openings, efforts were made to obtain average conditions found on pipe lines. Although swamps, bogs, river crossings, lakes, and other moist areas as well as oil soaked soils, salt washes, and other conditions represent trouble spots for the corrosion engineer, interest has been concentrated in the current project on average field conditions. Bacteriological and other analytical procedures used followed standard methods.

Results of Moisture Determinations

Water content of soils.

Moisture analyses of all the soils showed adequate water for microbial development. The data definitely show that a high moisture zone exists in the bottom of the pipe line ditch adjacent to the line.

Figure 4 summarizes soil moisture data for various types of soils indicating moisture content at the surface, in the semi-dense backfill above the pipe and ditch bottom samples. Free water was present in a number of bell holes at ditch bottom.

Moisture in Coating from Field Sampling.

Not only does the high moisture content of the ditch bottom indicate adequate water for bacterial growth but it also emphasizes the important role of moisture absorption by coating materials. The moisture present in the coating samples removed from pipe lines was determined by the Dean-Stark procedure ASTM-D95-46. This procedure makes use of controlled xylene distillation in a continuous apparatus with actual collections and measurement of the water present.

Interest in water absorption has led a number of producer and consumer laboratories to carry out tests for a number of years. Results have indicated that asphaltic materials take up water while coal tar does not.

Figure 5 summarizes the water content of coatings taken in the field and the water content of soil adjacent to the coating as well as the number of years the coatings had been in the ground. All samples of coal tar coating were low in water (less than 0.3 per cent) while asphaltic types of coatings varied with moisture present in the soil and the length of time buried. This field data confirms the reported dif-

ferences between coatings of different materials as shown by laboratory tests.

It is apparent from Figure 5 that from five to eight years of laboratory testing are necessary to secure reliable data on water penetration of coating materials.

Total Counts

Figures 6 and 7 record the aerobic, anaerobic bacteria count, soil moisture content and pH value of the soils at seven locations for each bell hole opened.

As expected the surface layers (Sample 1, see Figure 1) of the soil contain large numbers of bacteria. Most significant is the high bacterial population consistently present in the soil adjacent to the coating-wrapping systems (Sample 3). Counts here varied from a minimum of 320,000 per gram of soil to a maximum of 67,000,000. Less than one-tenth as many bacteria were present at the same depths in normal soil (Sample 7) where the counts varied from 2,000 to 2,000,000. These high populations of bacteria next to the pipe at ditch bottom suggest that the coating or wrapping materials serve as food material. This resembles the "rhizosphere" effect described near plant roots.

Hydrocarbon Utilizing Bacteria

In all locations studied typical hydrocarbon utilizing bacteria were found to be present. The types found were those commonly present in soils[8] and the numbers varied from less than one hundred per gram to several hundred thousands per gram. These microorganisms isolated were all bacterial types such as found by Harris, Kline and Crumpton[7] who reported asphalt utilizing bacteria were widely distributed. In fact distribution of hydrocarbons utilizing bacteria is probably world-wide since they have been isolated from soils in Germany,[8] Russia,[9] Japan,[10] Jordan[11] and many other countries as well as marine sediments from many locations.[6,12]

Figures 8 and 9 show typical rod-shaped bacteria such as *Pseudomonas aeruginosa.*

Figures 10 and 11 represent swollen rods or pleomorphic types of *Mycobacterium* species, while Figures 12 and 13 are electron microscope photographs of *Micrococcus paraffine* commonly found associated with asphaltic compounds in contact with the soil.[7]

Distribution of Aerobic Bacteria In The Pipe Line Ditch

An earlier report emphasized the finding of large numbers of aerobic bacteria in soil taken from the bottom of the pipe line ditch.[5] These high populations of aerobic forms indicate O_2 diffusion to pipe line levels. The presence of aerobic bacteria also leads to the conclusion that aerobic oxidation of coating materials may occur and also that O_2 may be a significant factor in pipe line corrosion following coating deterioraton.

Most emphasis in recent years has been given to the importance of sulfate-reducing bacteria in corrosion of bare metals following the work of Von Wolzgen Kuhr[13] and Starkey and Wight.[14] There is doubt that these forms attack hydrocarbons. Beerstecker[6] and recent publications have emphasized the importance of a mutual relationship between aerobic bacteria and the sulfate reducing anaerobes.[15] The presence of the large numbers of aerobic bacteria along pipe lines would greatly increase the possibilities for differential diffusion, concentration gradients, and conditions leading to electrolytic cell formation giving rise to corrosion by electrolysis.

This fact has not been unrecognized by workers in the field. For example, Hadley[16] stated that localized anaerobiosis would be expected to be of common occurrence. Results of three years of field sampling suggest that samplings in other areas of the United States should be developed. Such investigations are being incorporated into the Kansas State University project.

Main emphasis in bacteriological studies has been placed upon the *true* bacteria. Cultural conditions used were favorable for fungi and actinomyces. Large numbers of these were not observed as compared with the true bacteria. However, because of the known importance of other microorganisms, attempts are being made to evaluate all known forms of microscopic life in relationship to pipe line coating deterioration.

Acknowledgments

The author acknowledges the assistance given to this project by many of the operating gas and oil pipe line companies.

Summary

The soil environment of the undisturbed pipe line right-of-way was compared with the back-filled pipe line ditch. Results of the Kansas State University project show bacteria capable of growing on many types of coating materials. These bacteria have been found in large numbers adjacent to operating pipe lines. The significance of high moisture in soil adjacent to the lines, the moisture content of coatings and the presence of aerobic bacteria near the pipe lines were discussed.

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Any discussion of this article not published above will appear in the June, 1960 issue.

Electrical Resistance Measuring Device Plus Statistical Analysis Yield Unique Approach to Corrosion Problems*

By G. E. MOLLER and J. T. PATRICK

Abstract

A new technique for analyzing electrical resistance measuring device data is described. A 5 percent chromium, ½ percent molybdenum corrosion probe was used to measure corrosion rates in a refinery thermal cracking unit. By using the statistical technique of correlation by regression analysis, it was possible to determine which operational variables significantly affected corrosion rates and to calculate an equation to predict the corrosion rates in terms of these operating variables.

The linear regression equation which resulted from this analysis contained only the simple variable of sulfur content times velocity to predict corrosion rates. The corrosion rates calculated using this equation were found to compare favorably with the measured corrosion rates.

The statistical analysis of the corrosion rates measured by the corrosion probe resulted in: (1) Determining which operating variables significantly affected the corrosion rate, (2) Calculating an equation which can be used to predict corrosion rates under varying operating conditions.

Later, when made available, a standard program for the IBM 650 computer was used to perform the statistical calculations. The program used is called Stepwise Multiple Regression. It provides the advantages of processing a larger number of variables than with a hand calculator and eliminating an extensive knowledge of statistics. 2.1.1

Introduction

THIS PAPER presents a new technique for analyzing data collected from electrical resistance measuring device probes. The discussion describes the application of corrosion probes in an oil refinery processing unit. The corrosion data, when analyzed in the light of the factors suspected of promoting the corrosion, were found to reveal relationships which had been noted but heretofore not confirmed. The data collected were digested visually and then analyzed by statistical techniques. The calculation was done by hand until a program was found to perform these calculations on an electronic digital computer.

The purpose of this report is to introduce the application of the electrical resistance measuring device with these statistical techniques. This presentation describes an extension of the use of this instrument data in order to extract the maximum amount of useful information. It does not contribute anything new in the field of statistics and is not intended to be a text either in the basic use of the instrument or in the field of statistics.

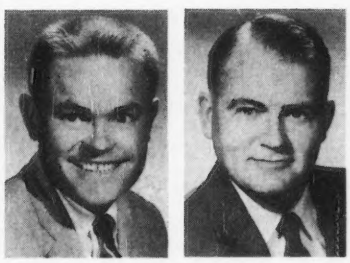
Use of Probes in Thermal Cracking Plant

Several corrosion probes were installed in a thermal cracking plant of the Union Oil Company of California at the Oleum Refinery. The purpose of these probe applications was to determine corrosion rates of various alloys in several of the most corrosive piping circuits. The information was to augment the corrosion rate data already accumulated by the older methods of measuring employing calipers, Vidigage, and Penetron. It was also used to obtain data on metals of greater alloy content not previously tested.

Figure 1 is a flow diagram of the thermal cracking unit. Attention will be devoted here to the results of two probes installed sequentially (timewise) in one location in the unit. The location (see Figure 1) was the suction header to two furnace feed pumps where the following conditions existed:

Line Size	12 Inches
Flow Rate, 15,000 B/D to Each Pump	
Temperature	740 F
Pressure	120 psi
Liquid	Gas oil containing combined organic sulfur, free sulfur, hydrogen sulfide, and some organic acids.

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Probes Used

The first probe studied was a high temperature, high pressure probe with an element of 5 percent chromium, ½ percent molybdenum, 40 mil diameter wire. The second probe was a high temperature, high pressure probe with an element of 9 percent chromium, 1 percent molybdenum wire.

When the plant was constructed, cracking coil tubing was carbon-molybdenum steel and 5 percent chromium alloy. Piping was carbon steel and 5 percent chromium. The main fractionating vessels, the fuel oil flash column, dephlegmator, and evaporator, were lined with 12 percent chromium. All cracking coil tubing and all cracking coil to fractionator transfer piping are presently 9 percent chromium. The fractionator to cracking coil charge piping is presently a mixture of 5 percent chromium, 9 percent chromium, and Type 304 stainless steel.

The 5 percent chromium probe was selected to aid in predicting the optimum life of the remaining 5 percent chromium. The 9 percent chromium probe was employed to augment knowledge of the newer 9 percent chromium system.

By examining Figure 2 the exact physical location of the probe may be noted. It was between Feed Pumps No. 2 and No. 3. When Feed Pump No. 3 was taken out of service and the spare was put on the line, the flow rate at the probe doubled.

Electrical resistance measuring device readings were taken daily, and sulfur content of the charge, flow rate, temperature at the probe, and temperature in the cracking coils were recorded daily.

5 Percent Chromium Probe

Figure 3 presents the results of the 5 percent chromium corrosion probe measurements. The cumulative corrosion measured by the probe is plotted on the vertical axis versus days of exposure on the horizontal axis. The curve starts at a low value on the left and proceeds to higher values with time. The slope of the curve represents corrosion rate. A nearly horizontal line is a low corrosion rate and a steep line is a high corrosion rate.

This plot indicated definite changes in corrosion rate. The changes were readily linked with operating changes in the plant. These latter changes have been noted on the graph. The lowest corrosion rates were experienced during low sulfur gas oil runs. Higher rates were experienced during high sulfur gas oil runs.

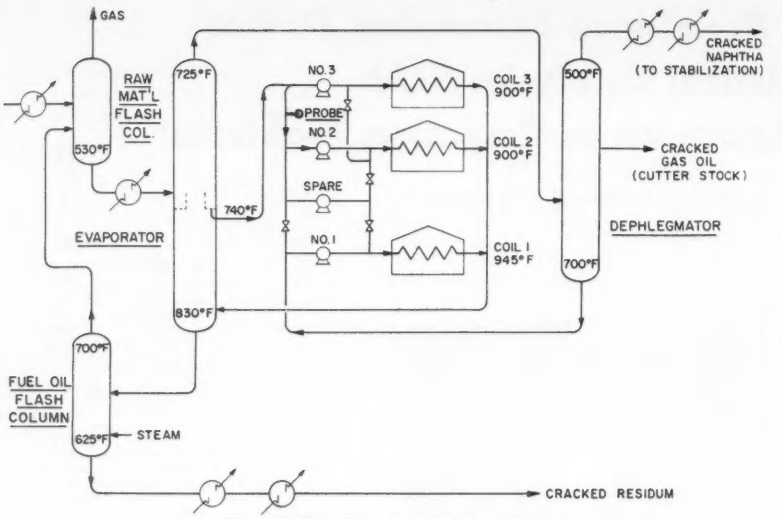


Figure 1—Flow diagram of thermal cracker.

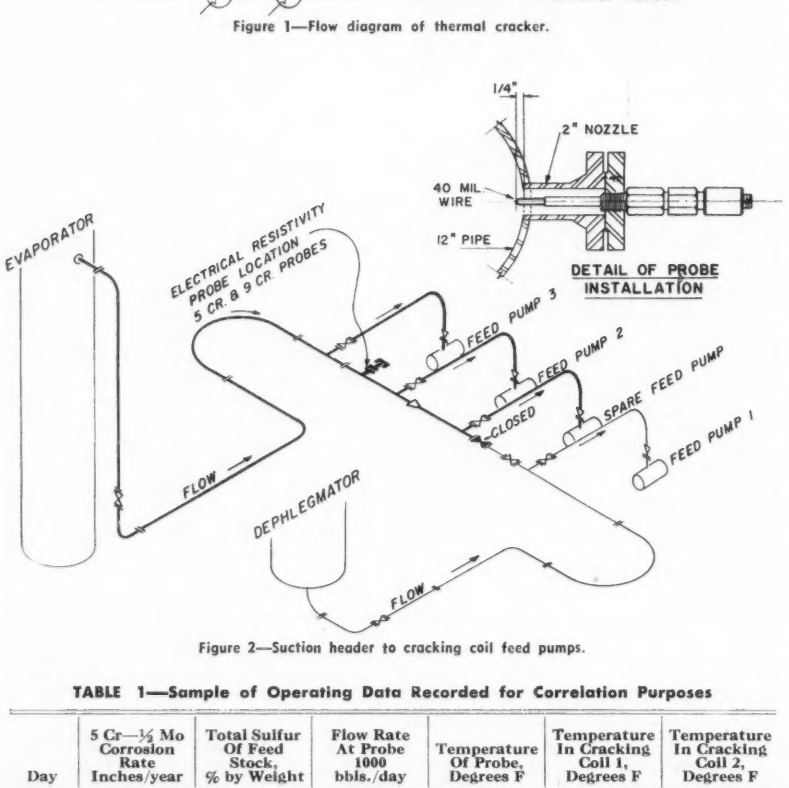


Figure 2—Suction header to cracking coil feed pumps.

TABLE 1—Sample of Operating Data Recorded for Correlation Purposes

Day	5 Cr—½ Mo Corrosion Rate Inches/year	Total Sulfur Of Feed Stock, % by Weight	Flow Rate At Probe 1000 bbls./day	Temperature Of Probe, Degrees F	Temperature In Cracking Coil 1, Degrees F	Temperature In Cracking Coil 2, Degrees F
1	0.117	4.1	16.9	753	922	885
2	0.107	4.1	17.0	748	925	885
3	0.088	4.0	17.1	749	925	886
4	0.077	4.1	16.6	747	925	887
5	0.091	4.2	17.0	745	934	895
6	0.040	0.8	17.5	743	940	905
7	0.048	0.7	35.0	762	936	904
8	0.022	0.8	34.5	760	935	895
9	0.077	4.1	33.8	752	928	887
10	0.121	4.1	33.6	754	928	887
11	0.143	4.4	33.2	749	930	887
...	...	...	...	...	...	...
...	...	...	...	...	...	...
...	...	...	...	...	...	...
40	0.122	4.1	14.5	730	936	884

The highest rate was logged during a high sulfur gas oil run when the flow velocity was doubled. To further test the effect of sulfur content, the low sulfur and high sulfur gas oils were mixed. This variation produced an intermediate corrosion rate.

The average daily corrosion rate of 290 microinches per day far exceeded the resolution of a 40 mil wire element of 4 microinches. It was, therefore, possible to calculate the corrosion rate in inches per year from the daily corrosion probe readings with little random error. A typical set of daily corrosion rates and operating data are illustrated in Table 1.

Figure 4 shows the corrosion rate and operating conditions at the probe plotted versus days of exposure. Figure 4 indicates some relationship between the operating conditions and the corrosion rate but the effects cannot be determined quantitatively because several variables are changing simultaneously. However, the following observations can be readily made. On the fifth day the sulfur content of the unit charge dropped from 4.2 percent to 0.8 percent, which resulted in a lowering of the corrosion rate from 0.09 inch per year to 0.04 inch per year. Corrosion rates remained low throughout the three-day low sulfur gas oil operation.

Figure 4 indicates a lag of about twelve hours before the change in operating conditions appeared as a change in corrosion rate. On the fifth day the spare pump was started, doubling the fluid velocity. This change did not immediately affect the corrosion rate but after the eighth day, when coupled with a return to high sulfur gas oil, it pushed the corrosion rate to a new high of 0.23 inch per year. Apparently, velocity or turbulence has its effect but it is not as pronounced as the sulfur effect, at least in the relative magnitudes of changes experienced in this test. On the twenty-seventh day, the low and high sulfur gas oils were combined lowering the total sulfur of the charge to 3.2 percent resulting in a corrosion rate of 0.07 inch per year.

The temperature at the probe was also plotted. The purpose of this plot was to determine if a relationship existed between temperature and corrosion rate. No correlation was visible in the narrow range covered by the operating temperatures.

Description of Regression Analysis

Regression analysis was employed to test the observation that corrosion rate is dependent on sulfur content of the gas oil and is affected by the velocity of the flowing medium. It was also used to determine if temperature were actually significant although it was not evident by visual observation.

Regression analysis is used for the correlation of data. It is applied in many fields: physical chemistry, agriculture, social science, operations research, etc. Recently, engineers, because of increasing amounts of engineering data to be processed, have resorted to statistical analysis and correlation studies to aid them in achieving the best possible results from their work.

The rest of this section deals with regression analysis. Those who wish to explore the subject further may find Volk's[1] work of interest.

Regression analysis is used to determine if there is a correlation between one dependent variable and one or more independent variables. The result is a linear regression in the case of one in-

dependent variable, and a multiple linear regression in the case of more than one independent variable.

A linear regression is a straight line, the best line that can be plotted through data described by two variables, one dependent and one independent. The form of the regression is an algebraic expression for the line as shown below:

$$Y = a + bx \quad (1)$$

Where:

Y = The Dependent Variable
x = The Independent Variable
a = Constant of the Equation, Y Intercept
b = Regression Coefficient of the Equation, Slope

A multiple linear regression is a plane on a three dimensional coordinate system, for the case of one dependent and two independent variables, or a multi-dimensional plane in the case of more variables. Again, an algebraic expression is produced from the analysis with the form shown below. It contains a linear combination of independent variables.

$$Y = a + bx_1 + cx_2 + \ldots\ldots nx_n \quad (2)$$

Where: x_1, x_2, x_n Are Independent Variables

The second feature of this technique permits results to be tested for reliability. It can be used to determine whether the correlation, if there is one, is a result of pure chance or whether there is a true interrelationship of cause and effect. It can be used to determine how significant each variable is and how much each contributes to the relationship. It can be used also to determine what confidence limits can be placed on the relationship, and how much deviation or error can be expected.

Simple correlation studies can be performed mathematically on a hand calculator if the study involves two or three variables. More complicated situations with as many as 33 variables can be handled on an electronic data processing machine. The advantage of the computer is that it permits programs to be written without requiring extensive knowledge by the user of the theory of statistics. The instructions for these programs explain the listing of the data and the setting up of the program for transcription to a data processing center. They also explain how to interpret the answers. Although several programs have been written for the IBM 650, the Stepwise Multiple Regression Program,[2] which was used to evaluate the authors' data, avoids most of the difficulties encountered in regression analysis.

An additional program has been written for the variables of the Stepwise Multiple Regression Program to test them as curvilinear functions.[3] Data often fit a curve better than a straight line. With this program, it is helpful to know the theoretical relationship (curve) between the dependent and independent variables. The program does not determine which function (curve) should be used but only confirms that the relationship selected by the engineer fits the data and with what significance it fits.

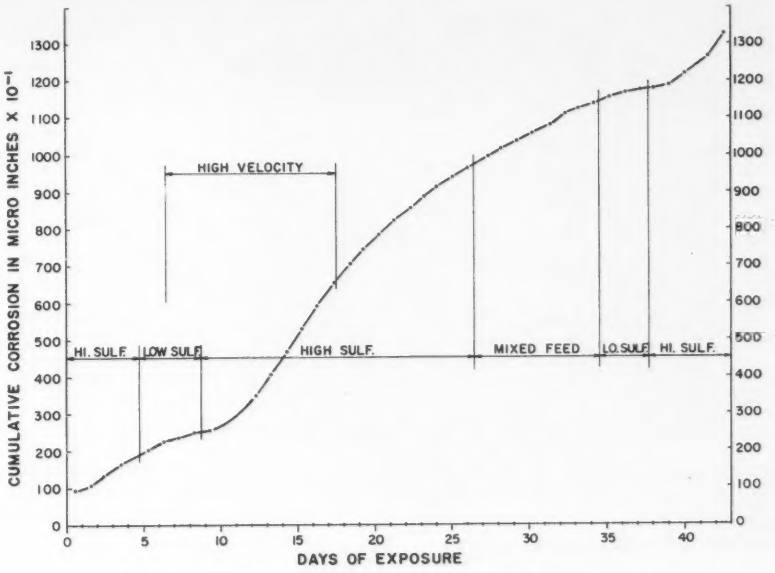


Figure 3—Cumulative corrosion of 5 Cr-½ Mo electrical resistivity probe.

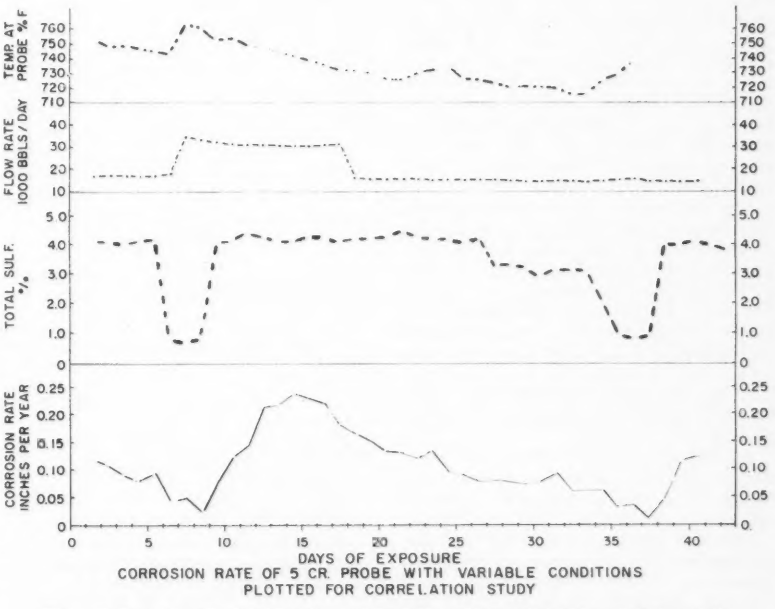


Figure 4—Corrosion rate of 5 Cr probe with variable conditions plotted for correlation study.

Statistical Analysis of 5 Percent Chromium Probe

The statistical analysis of the corrosion rates of the 5 percent chromium probe was first performed by hand calculation because the existence of the Stepwise Multiple Regression Program was not known. Since it was observed in Figure 4 that sulfur and velocity seemed to be the operating variables principally affecting the corrosion rate, the first calculation involved only these two variables.

Figure 5 indicates the suspected dependence of corrosion rate on sulfur and velocity. To reduce the amount of calculation involved, seven points were chosen which covered a wide variation of sulfur and velocity. These points are shown in Figure 6.

Two equations were calculated by hand, using sulfur and velocity as independent variables. In the first equation it was assumed that the corrosion rate was a linear function of both sulfur and velocity. The following equation resulted:

$$Y = 0.010 + 0.032S + 0.042V \quad (3)$$

Where:

Y = Corrosion Rate, in Inches Per Year
S = Sulfur Content, in Per Cent
V = Velocity, in Feet Per Second

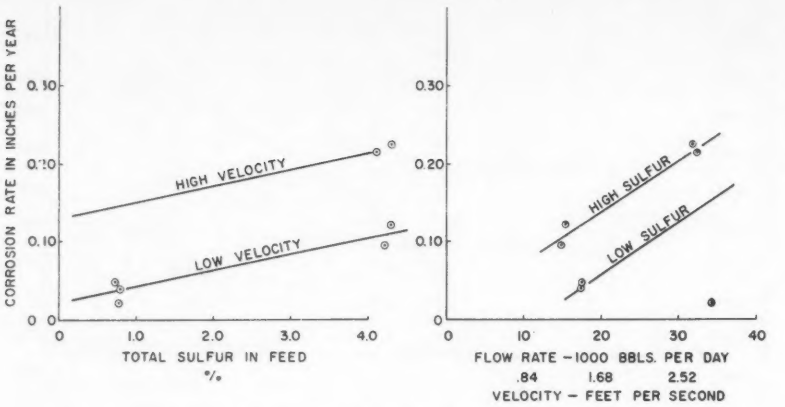


Figure 5—Plots of corrosion rate vs total sulfur and flow rate (velocity).

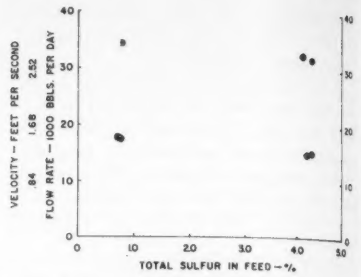


Figure 6—Independent variables—plot of flow rate (velocity) vs total sulfur in feed.

In the second equation it was assumed that the corrosion rate was a linear function of the product of sulfur and velocity and the following equation resulted:

$$Y = 0.010 + 0.018SV \qquad (4)$$

Equation 4 is closer to the true relationship between the variables, since Equation 4 was found to be much more significant. The significance of the two equations was tested by the technique known as analysis of variance. This powerful tool disclosed that Equation 4 was well above 99 percent significant whereas Equation 3 was below 95 percent significant. Equation 4 was accepted as the result. The only peculiar aspect is that both equations indicate that there is corrosion when both sulfur and velocity are zero. However, the point of zero sulfur and zero velocity was not within the area of the data investigated and prediction of this point would require extrapolation which can lead to large errors. It is possible that corrosion rates are affected by a different law when velocity passes from turbulent to streamlined flow.

In Figure 7 the corrosion rate predicted by Equation 4 is compared with the probe corrosion rate. The excellent correlation is evident. The only places where the two curves diverge are immediately following an abrupt change in operation conditions, particularly flow rate. It probably takes time for the higher rate to erase the protective film generated at the lower rate. Figure 8 is a plot of corrosion rate versus the factor combining velocity and sulfur, the significant independent variable derived from the regression analysis. The various data points were obtained from the experimental data as shown in Table 1. On this plot is drawn the best line through the data calculated from the derived Equation 4. It was this line with its attendant equation that the regression analysis was performed.

Since a function of sulfur and velocity was used as a term in Equation 4, this equation exhibits nonlinear characteristics when corrosion rate is plotted in three dimensions as shown in Figure 9. Equation 4 shows that corrosion rate varies directly with velocity at constant sulfur and directly with sulfur at fixed velocity. Corrosion of the system is a function of the sulfur throughput and cannot be circumvented by mixing feeds of low and high sulfur gas oil. It can only be reduced by lowering sulfur content of the feed stock.

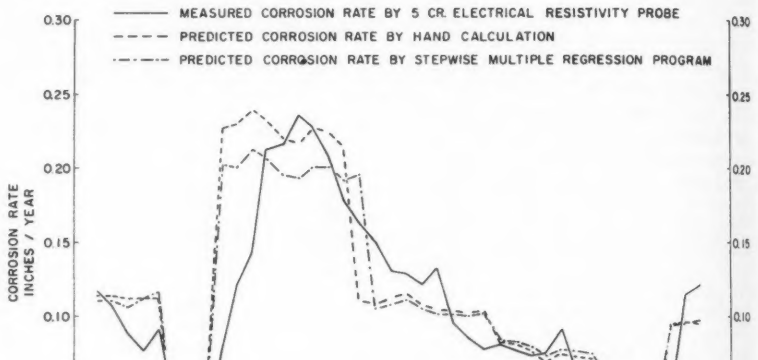


Figure 7—Corrosion rate of probe compared with regression analysis predicted corrosion rates.

TABLE 2—Eleven Variables

OPERATING VARIABLES	UNITS
Sulfur content of feed (S)	% by weight
Velocity of stream (V)	feet per second
Temperature of probe (T_1)	°F
Temperature in cracking coil 1 (T_2)	°F
Temperature in cracking coil 2 (T_3)	°F
FUNCTION OF OPERATING VARIABLES (INDEPENDENT VARIABLES)	**NUMBER ON PRINT OUT SHEET**
S	1
S^2	2
S^3	3
V	4
S x V	5
S x V^2	6
T_1	7
T_2	8
T_3	9
e^{T_1}	10
$e^{\frac{125.73}{T_1 + 460}}$	..
DEPENDENT VARIABLE	
Y (Corrosion Rate) inches per year	11

The corrosion data for the 5 percent chromium probe were next analyzed using the Stepwise Multiple Regression Program and the IBM 650 computer. (It should be noted that all work up to this point was performed on a hand calculator.) Thirty-seven sets of data

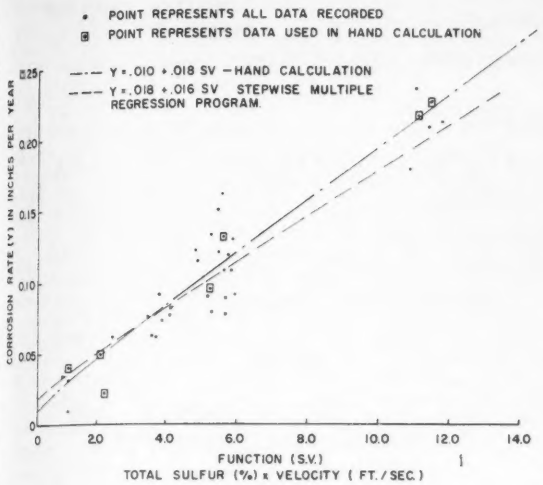


Figure 8—Best line through data—corrosion rate vs function (SV).

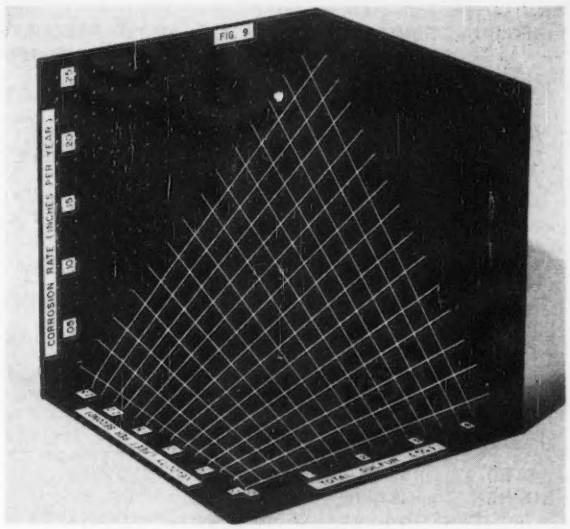


Figure 9—Corrosion rate plotted in three dimensions.

were used instead of seven sets as in the case of the hand calculation.

In addition to the calculations performed by hand, the Stepwise Multiple Regression Program permits ony significant variables to enter the final equation. The eleven variables shown in Table 2 were entered into the computer as independent variables. As shown in the listing, functions representing straight lines, power curves, hyperbolic surfaces, exponentials, and the temperature reaction rate were tested. The print-out sheet for the program, Figure 10, describes the correlation. The function SV (Variable 5) was the most significant variable of the eleven entered into the computer. The final equation was:

$$Y\,(\text{Corrosion Rate}) = 0.0178 + 0.0159SV \qquad (5)$$

Coefficients shown here differ from those on the print-out sheet because the corrosion rates were scaled by a factor of ten.

Besides the final regression equation, there is additional statistical information included in the results of the Stepwise Multiple Regression Program. Much of this information is beyond the scope of this discussion. However, two items are of interest. They are:

1. The standard deviation of the dependent variable.
2. The tabulation of the actual and predicted corrosion rates and the difference between these for each data set.

The standard deviation of the dependent variable describes the expected error when using the regression equation. Ninety-five percent of all corrosion data is expected to fall within ± two standard deviations from the prediction. The standard deviation of the equation for predicting the corrosion rate of 5 percent chromium was 0.0232 inch per year. The standard deviation of "Y" is the error in the experiment which is not accounted for by the correlation. It includes experimental error plus other factors that may contribute to the corrosion process. The close proximity of the actual and predicted corrosion rates can be seen in the tabulation of the results in Figures 7, 8, and 10.

Although temperatures were premitted to enter the predicting equation, no temperature term appeared in the final equation. The reason for this is readily evident when it is found that the absolute temperature varied less than 3 percent. The corrosion rates were, therefore, measured at essentially a constant temperature. If an experiment could be planned with a wider temperature spread, it is felt that an expression could be produced including temperature effects. Probes at higher temperatures in the same stream have been tried, but to date a probe element has not been produced that is rugged enough to withstand these elevated temperatures.

Statistical Analysis of 9 Percent Chromium Probe Data

The corrosion rates obtained from the 9 percent chromium probe were also analyzed by using the Stepwise Multiple Regression Program. The following equation resulted:

$$Y = 0.007 + 0.007S \qquad (6)$$

Where:

Y = Corrosion Rate, in inches per year

S = Sulfur Content, in percent

However, the standard deviation for this equation is 0.013 inch per year. Since the average corrosion rate was 0.029 inch per year, it can be seen that the equation is not too significant. The high standard deviation of the final equation is caused primarily by the error in measuring the corrosion rate of the 9 percent chromium probe daily. The average corrosion rate of 75 microinches per day was low enough so that absolute resolution of 4 microinches could cause appreciable random error.

TABLE 3—List of Equations

1. **Linear Regression** $Y = a + bx$
   Where: Y = dependent variable
   X = independent variable
   a = Y intercept, a constant of the equation
   b = slope, regression coefficient
2. **Multiple Linear Regression** $Y = a + bx_1 + cx_2 + \ldots nx_n$
   Where: x_1, x_2 & x_n = independent variables
   a = Y intercept, a constant
   b, c & n = slopes, regression coefficients
3. **First Model** $Y = a + bS + cV$
   (Hand)
   Where: Y = corrosion rate, inches per year
   a, b, c = constants
   S = total sulfur, %
   V = velocity, feet per second
   with resulting equation
   $Y = 0.010 + 0.032S + 0.042V$
4. **Second Model** $Y = a + bSV$
   (Hand) with resulting equation
   $Y = 0.010 + 0.018\ SV$
5. **Stepwise Multiple Regression Output**
   (Computer) **5% Chromium**
   $Y = 0.0178 + 0.0159\ SV$
6. **Stepwise Multiple Regression Output**
   (Computer) **9% Chromium**
   $Y = 0.007 + 0.007S$

Statistical Analysis Discussion

The corrosion rates registered by the probes and the results of the regression analysis are in good agreement with plant experience. There has been an upward trend in the sulfur content of the feed stocks. With this trend, more severe corrosion rates have been encountered. This increase has dictated the change to 9 percent chromium and Type 304 stainless steel piping. Evidence of the velocity effect is substantiated by the higher corrosion rates found in the smaller diameter discharge piping where the velocities are greater. The absolute values of corrosion rate from this study tend to be high in straight runs of piping by a factor of 1.5.

Caution should be used when interpreting corrosion probe data[4]. Probes represent the corrosion rate at the point of insertion but do not always duplicate it. In this case the local turbulence at the wire protruding into the line is greater than the turbulence at the pipe

ABBREVIATED COPY OF COMPUTER PRINTOUT SHEET

RESIDUAL SUMS OF SQUARES AND CROSS PRODUCTS
Y IS CODED AS THE HIGHEST NUMBERED VARIABLE

1	101	58.00000+	102	298.64700+	103	1324.21170+	104	8.75380+

ETC.

AVERAGE VALUE OF VARIABLES
Y IS CODED AS THE HIGHEST NUMBERED VARIABLE

1	100	3.38324+	200	13.01390+	300	52.10074+	400	1.60189+
2	500	5.65618+	600	22.26725+	700	.34459+	800	.32865+
3	900	.88432+	1000	1.42160+	1100	1.07730+		

SIMPLE PARTIAL CORRELATION COEFFICIENTS OF VARIABLES
Y IS CODED AS THE HIGHEST NUMBERED VARIABLE

1	102	.98660+	103	.95717+	104	.32545+	105	73699+

ETC.

CONSTANT A OF REGRESSION

1.07730+

STEP NUMBER	F VALUE	VARIABLE ENTERING	STD. DEV. OF Y
1	199.48950+	5	.23230+

F value used for significance test. This value well above 99 % significant.

Variable 5, Table 2

REGRESSION COEFFICIENTS

VARIABLE NUMBER		COEFFICIENT
1	5	.15898+

b of Eq. 5, Table 3, Slope — regression coefficient

STANDARD DEVIATION OF REGRESSION COEFFICIENTS

		STANDARD DEVIATION
1	5	.01125+

CONSTANT A OF REGRESSION

1	.17808+

a of Eq. 5, Table 3, Y intercept — constant of resulting equation.

INVERSE MATRIX OF PARTIAL CORRELATION MATRIX FOR VARIABLES INCLUDED IN THE REGRESSION EQUATION

505	1.00000+

VARIABILITY OF VARIABLES NOT IN REGRESSION EQUATION
(FRACTION UNACCOUNTED FOR BY THE EQUATION)

VARIABLE NUMBER	FRACTION VARIABILITY	VARIABLE NUMBER	FRACTION VARIABILITY
1	.45684+	7	.95668+
2	.41886+	8	.47389+
3	.40880+	9	.73217+
4	.23044+	10	.96898+
6	.00865+		

Therefore, resulting equation from stepwise multiple regression is:

$$10Y = 0.178 + 0.159\ SV^{*}$$

(Eq. 5, Table 3)

* Input corrosion rates were multiplied by 10 for ease of computer handling. Results must be divided by 10.

COMPARISON OF PREDICTED VALUES WITH ACTUAL VALUES

DATA SET	ACTUAL VALUE	PREDICTED VALUE	DIFFERENCE (ACT.– PRED)
1	1.17000+	1.09714+	.07286+
2	1.07000+	1.10366+	.03366–
3	.88000+	1.06471+	.18471–
4	.77000+	1.12624+	.35624–
5	.91000+	1.12624+	.21624–
6	.40000+	.35681+	.04319+
7	.48000+	.34514+	.13486+
8	.22000+	.35558+	.13558–
9	1.43000+	2.11573+	.68573–
10	2.12000+	2.05802+	.06198+
11	2.16000+	1.95102+	.20898+
36	1.14000+	.95390+	.18610+
37	1.22000+	.96678+	.25322+

When comparing Data Set Numbers with Days on Table 1 and Figs. 3, 4, 7 & 8, it will be found that data for days 9, 10 & 38 had been left out to eliminate lag effect.

NOTES

Figure 10—Stepwise multiple regression results.

wall which results in accelerated corrosion of the wire. Corrosion rates measured by the probe more nearly duplicate severity observed in valve bodies and other pipe fittings where the turbulence was approximately the same as at the probe.

It should be pointed out that it is dangerous to extrapolate the regression equations beyond the limits of the data used. The correlation is good for the span of the data but it may become inaccurate at some point far beyond. In this case, the velocities used were quite low, in the range of 1.4 to 2.8 feet per second. It may be that velocity versus corrosion rate is some curved relation but in this example only a small portion of the curve was tested, so short that it is linear in this portion. Also, to detect a curvilinear relationship, three widely spaced values of the independent variable are required. Only two points were tested in this case for the independent variable of velocity. Three widely spaced values of sulfur were tested, however. The sulfur effect did not prove to be curvilinear over a wide range.

For those who wish to explore statistical analysis further, a reference[1] is listed in the bibliography which gives additional information on the statistical techniques of analysis of variance and regression analysis which were used in analyzing the data presented herein.

Conclusions

In conclusion, the authors admit that this work is just a starter. The method has produced a usable empirical formula for the corrosion rate of a single alloy. The temperature of the experiment did not vary enough to show its effect. A wide spread of feed sulfur contents and a satisfactory spread of velocities were available for inclusion in the work. Corrosion rates calculated by the derived formula show satisfactory correlation with actual plant data.

This technique is a tool by which the corrosion engineer can obtain a better understanding of the individual effects that promote corrosion and should enable him:

1. To determine the effect operating variables have on corrosion rates.
2. To permit him to talk of corrosion cost penalties when discussing proposed operational changes.
3. To predict the remaining life of equipment under varying operating conditions.
4. To provide a good basis on which to select materials of construction for new and replacement facilities to permit a satisfactory financial payout.

Much work remains to be done. Experimental data encompassing a wider variation of operating variables are required. To this could be added corrosion rate data on different alloys and the effects of other corrodents beside sulfur. If sufficient data were available, it appears possible that a universal formula could be derived for the corrosion effect. This may appear fantastic at the present time, but the necessary tools and techniques are available.

Acknowledgment

The authors are indebted to Dr. D. J. Wilde of the Univ. of California at Berkeley for his help in the field of statistical analysis.

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Any discussion of this article not published above will appear in the June, 1960 issue.

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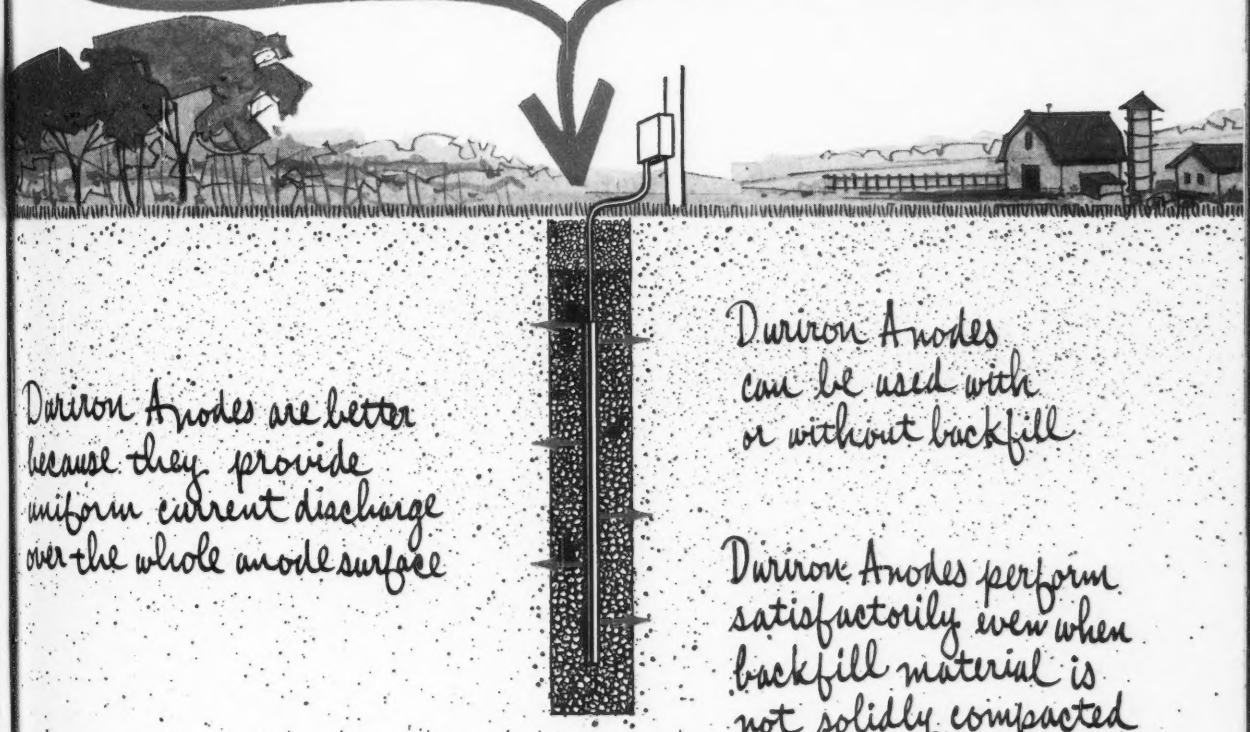
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*S106		Oil			188° F.	
S107		Oil			140° F.	
S108		Oil			280° F.	
330	Standard	Water or Brine		10° F.		
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206	Standard	Fresh Water	Brine	5° F.		
207	Standard	Oil	Fresh Water	— 5° F.		
208	Standard	Oil		—15° F.		
209	Standard		Water	—60° F.		
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